

UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

THE BOEING COMPANY

Petitioner,

v.

THE REGENTS OF THE UNIVERSITY OF CALIFORNIA

Patent Owner.

Case IPR2024-00761

Patent 7,487,684

**PATENT OWNER'S PRELIMINARY RESPONSE
PURSUANT TO 37 C.F.R. § 42.107(a)**

TABLE OF CONTENTS

I.	SUMMARY OF ARGUMENT.....	1
	A. Deficiencies of Grounds Challenging the Claims.....	2
	A. Wang-Dissertation (Ex. 1005)	6
	a. Silicon Substrate Exhibit Damage Patterns	9
	b. Fused Silica Substrates Do Not Exhibit Damage	12
	B. Gupta-1992 (Ex. 1011).....	13
	C. Wang-2002 (Ex. 1011)	18
III.	THE '684 PATENT	21
IV.	CLAIM CONSTRUCTION	25
V.	A PERSON OF ORDINARY SKILL IN THE ART	26
VI.	THE BOARD SHOULD DENY INSTITUTION BECAUSE THE PETITION FAILS TO ESTABLISH A LIKELIHOOD OF SUCCESS FOR ALL GROUNDS ASSERTED FOR INDEPENDENT CLAIMS 1 AND 13	26
	A. Ground 1 – Anticipation By Wang-Dissertation.....	26
	1. The Petition Fails to Establish Public Availability of the Wang-Dissertation.....	26
	2. The Petition’s Anticipation Contention	32
	3. The Wang-Dissertation Fails to Anticipate the Independent Claims	34
	4. The Wang-Dissertation Fails to Enable a Glass Element and Substrate Coupled Structure	37
	a. Significant Problems in Tested Silicon Substrates as Compared to Fused Silica Substrates Support Non-Enablement.....	38
	b. Evidence in the Wang-Dissertation Logically Raises Concerns Regarding Substrate Damage When Coupled with Fused Silica	39
	c. Silicon Substrates Exhibit Damage at Relatively Low Tensile Stresses Well Below Those Achieved with Fused Silica as the Substrate.....	40
	d. Fused Silica produces Much Higher Tensile Stresses Further Inducing Damage in a Coupled Substrate.....	41

e.	Substrate Attenuation Significantly Impacts Test Interface Stress	43
f.	Boeing’s Expert was Informed on Enablement But Failed to Provide Any Support or Evidence Showing the Future Work was Enabled	46
5.	Conclusion - The Wang Dissertation Fails to Enable One of Skill in the Art to Make and Use a Fused Silica and Additional Substrate Combination	47
B.	Ground 4 – Anticipation by Gupta-1992	48
1.	Gupta-1992 Fails to Describe a Glass Element that Propagates and Modifies a Stress Wave	52
2.	The Petition Fails to Provide Any Support for the Contention that Fused Quartz and Fused Silica Have the Same Properties and the Wang-Dissertation Supports the Opposite Conclusion	54
C.	Ground 3 – Wang-Dissertation in View of Gupta-1992.....	56
1.	The Rationales Provided in the Petition	58
2.	The Wang-Dissertation Does Not Enable Using Fused Silica in Combination with a Substrate to Test Interfacial Strength.....	60
a.	The Wang-Dissertation Only Described Substantial Loading at the Test Interface without Damage When Using a Single Fused Silica Substrate.....	62
b.	The Petition’s Reliance on Gupta-1992 Ignores the Failed Results of the Relied on Configuration	65
D.	Ground 7 – Wang-2002 in view of Gupta-1992	68
1.	The Rationales Provided in the Petition	70
2.	Considering the Art as a Whole Neither Wang-2002 Nor Gupta-1992 Provide Support for Petitioner’s Rationales	71
a.	Significant Problems in Tested Silicon Substrates as Compared to Fused Silica Substrates	73
b.	Silicon Substrates Exhibit Damage at Relatively Low Tensile Stress Without Fused Silica	74
c.	Fused Silica produces Much Higher Tensile Stresses Further Inducing Damage in a Coupled Substrate	75
d.	Substrate Attenuation Significantly Impacts Test Interface Stress	77

e. Gupta-1992 - Substrate Damage	79
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TABLE OF EXHIBITS

Exhibit	Description
2001	“Glass-modified stress waves for adhesion measurement of ultra thin films for device applications,” Gupta et al.

I. SUMMARY OF ARGUMENT

The Board should deny institution of The Boeing Company's ("Boeing") Petition because it fails to establish a reasonable likelihood of success with respect to proposed Grounds 1-7. In this Patent Owner Preliminary Response, Patent Owner shows the deficiencies of Grounds 1, 3, 4 and 7, which are the only grounds that implicate independent claims 1 and 13. All other asserted Grounds are necessarily deficient.

The technology of U.S. Patent 7,487,684 ("the '684 Patent") relates to the use of laser spallation to generate a tensile stress between a substrate and a coating. Ex. 1001, Abstract. The tensile stress, if of sufficient amplitude, separates the coating from the substrate. The purpose of this tensile stress is to separate the coating from the substrate to indicate the tensile strength of the substrate/coating interface. Ex. 1001, 2:1-8. In operation, a structure including a constraining layer, an energy absorbing layer, a glass substrate element, a substrate and a coating, is ablated with a laser directed through the constraining layer to strike the energy absorbing layer. Ex. 1001, FIG. 1. Absorption of the laser energy leads to a sudden expansion of the energy absorbing layer that generates a compressive shock wave directed towards the test coating interface. *Id.* at 2:25-29. The reflection of this compression pulse

from the free surface of the coating becomes a tension pulse that can lead to the removal of the coating if of a sufficiently high amplitude. *Id.* at 2:31-34.

In the Wang-Dissertation, various substrates were tested for use with laser spallation, including fused silica and silicon where either the fused silica or silicon was bonded directly to the coating layer. *See generally, e.g.*, Exs. 1005 and 1011. A primary problem with the silicon substrates was damage experienced during testing. As noted by the Wang-Dissertation, the silicon substrate attenuates stress and the silicon substrates exhibited damage patterns, which are typical for silicon substrates and not desired in laser spallation experiments as they result in causing the test film to come off the substrate before the interface fails. Ex. 1005, pp. 47, 61.

A. Deficiencies of Grounds Challenging the Claims

Grounds 1, 3, 4 and 7 rely on four different references either alone or in combination: (1) the Wang-Dissertation (Ex. 1005), (2) Gupta-1992 (Ex. 1011); (3) Wang-2002 (Ex. 1016); and (4) the journal article from Barker, L.M., Hollenbach, R.E. titled “Shock Wave Studies of PMMA, Fused Silica, and Sapphire” (Ex. 1021). None of the these references show all of the features recited in claims 1 and 13 as they fail to show the use of a “glass element” that is configured to propagate and modify a stress wave to generate a tensile stress between the substrate and the coating **in combination with** another substrate disposed between the “glass element” and the test coating.

Wang-Dissertation Deficiencies

Absent is any likelihood of success in relying on the Wang-Dissertation as Boeing fails to establish the public availability of this reference, oddly arriving at a date one year and a day prior to the earliest priority date of the '684 Patent. If 102(a) were to be implicated, earlier invention could be shown in “Glass-modified stress waves for adhesion measurement of ultra thin films for device applications,” authored by Gupta et al. Ex. 2001.

Aside from this deficiency, the Wang-Dissertation fails to support either anticipation or obviousness as it only describes and enables a POSITA to use either a silicon substrate (which experiences substrate damage and low interface stresses), or alternatively, the intrinsically strong fused silica substrate bonded directly to the coating when using laser spallation to test thin-film adhesion. To cure this deficiency, Boeing relies on a statement in the last paragraph of the Wang-Dissertation under the section title “Future Work” that provides:

By inserting an extra layer of the desired substrate material between the fused silica and the thin film, the current technique can be used to test a variety of substrates and films, especially those of interest to the microelectronics industry, such as polyamide, copper (Cu), aluminum (aluminum) and polysilicon on silicon substrates.

Ex. 1005, p. 124

This contemplation of future work is never enabled within the Wang-Dissertation as the clear evidence of record establishes critical problems of substrate damage and

substrate attenuation. The Wang-Dissertation emphasizes that this stress attenuation is even greater with shock waves produced by the fused silica substrate. Ex. 1005, p. 38. Accordingly, this stand-alone contemplation of future work fails to lead a POSITA to expect successful results when placing a silicon substrate downstream of the fused silica in view of the clear and consistent results showing undesired substrate damage, and substrate attenuation that is even greater with fused silica shock waves. These deficiencies show the Petition fails to establish a likelihood of success for Grounds 1, 2 and 3.

Gupta-1992 and the Barker and Hollenbach Article

Boeing relies on Gupta-1992 in grounds based on both anticipation (Ground 4) and in obvious grounds (Ground 3, 5, 6 and 7). The primary deficiency in Boeing's reliance on Gupta-1992 is its contention that it uses fused quartz that allegedly teaches a glass element that propagates and modifies a stress wave. Absent from Gupta-1992 is any evidence that the fused quartz exhibits this property. Even Boeing's expert concedes Gupta-1992 does not describe its "intended function and scientific mechanisms." Ex. 1003, p. 79. To provide support for these properties, the Petition conflates the terms and materials "fused silica" and "fused quartz" and even cites to the Barker and Hollenbach article for support. Ex. 1021. This correlation fails as the Barker and Hollenbach article only describes the properties of high-purity fused-silica, not fused quartz. Ex. 1021, p. 4209 (2nd column, first full

paragraph). This conflating fails as the Wang-Dissertation thoroughly discusses and cites to Gupta-1992, yet concludes “[a]though shock development in fused silica was demonstrated in the 1970s by Barker and Hallenbach using a gas-gun facility, the usefulness of such shocks in applications to laser spallation has not been reported.” Ex. 1005, p. 81; see p. 4, 8 9, 22, 40 and 42 for citations to Gupta-1992. Otherwise, in several other instances the Petition relies on the structure of Gupta-1992 in which fused quartz is used in laser spallation. Glaringly, the Petition ignores that this configuration resulted in damage to the underlying Pitch-55 substrate—a result that is not desired in laser spallation interfacial strength testing as the substrate damage prevents testing of the interface strength. These deficiencies show the Petition fails to establish a likelihood of success for Grounds 3-7.

Wang-2002

Wang-2002 is a repeat and less detailed disclosure of the material in the Wang-Dissertation. Absent is in this paper is any “Future Work” comments or use of fused silica in combination with an intervening substrate. To remedy the deficiencies of Wang-2002, the Petition seeks to modify Wang-2002 based on the structure of the failed test structure from Gupta-1992, which clearly belies any expectation of success. The rationales provided by the Petition and the accompanying expert declaration ignore these clear failures in Gupta-1992 when using this structure and fail to provide any reasoning to overcome those failures.

These deficiencies show the Petition fails to establish a likelihood of success for Ground 7.

As is outlined in detail within this POPR, Petitioner submits that the Petition fails to show a likelihood of success with respect to independent claims 1 and 13 addressed in grounds 1, 3, 4 and 7 because none of the cited references discloses or suggests the glass element of those claims, or its function. Accordingly, institution of this Petition should be denied.

II. BACKGROUND OF THE ART

A. Wang-Dissertation (Ex. 1005)

1. Introduction

The Wang-Dissertation generally relates to thin-film adhesion measurement by laser-induced stress waves. The techniques rely on using a thin absorbing layer confined between a confining plate and a back surface of a substrate. Ex. 1005, p. 3, 7. The apparatus applied by Wang during investigations of laser-induced stress waves is shown below in FIG. 2.1.

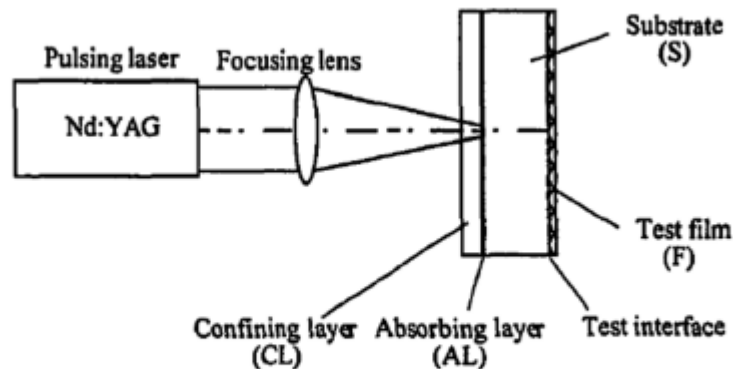


Figure 2.1: Schematic of the tensile laser-spallation technique.

The Schematic above shows from the laser impingement side, a confining layer (CL), an absorbing layer (AL), a substrate (S) and a test film (F). A test interface lies between the absorbing layer (AL) and the test film (F). Ex. 1005, p. 7-8. Wang's project sought to determine the strength of "thin-film" interfaces by producing tensile stress at the test film interface. *Id.* In effect, laser energy impinging the adsorbing layer confined by the substrate and the confining layer induces a compression wave in the substrate. The compression wave propagates through the test film/substrate interface and is reflected back from the free surface of the test film as a tension wave that loads the test interface under tension. *Id.* However, due to the release of stress at the test film surface, the stress realized at the test interface is "much lower than the stress in the substrate." *Id.*

2. Testing of Fused Silica as the Substrate

To understand the effect of substrate material on the generation of interface tensile stress, a fused silica substrate and a silicon substrate were selected in the Wang-Dissertation. Ex. 1005, p. 7. Wang recognized prior art showed fused silica exhibits non-linear properties of elasticity that soften under compression, and because of that the fused silica may potentially develop a shock that in turn can enhanced loading at the film interface. Ex. 1005, p. 7. Consequently, Dr. Wang studied the use of a fused silica substrate as a material to overcome these limitations as compared to an silicon substrate.

3. Problems Inhibiting Tensile Stress Formation at the Test Interface and Measuring Adhesion Strength

The Wang-Dissertation identifies several problems inhibiting adequate tensile stress at the test interface and determining thin-film adhesion: (1) the finite size of the YAG laser beam; (2) attenuation of stress due to the thickness of the substrate; (3) realized tensile stresses at the test interface being lower than the stress acting on the substrate as the free surface of the test film releases some of the stress; and (4) damage to the substrate. Ex. 1005, pp. 7-8, 21-22 and 46-51.

4. Tensile Spallation Experiments - Silicon and Fused Silica Each Tested as Substrates

Junlan Wang performed experiments on both a bare silicon substrate and a fused silica substrate having the general structure shown in FIG. 3.2 below.

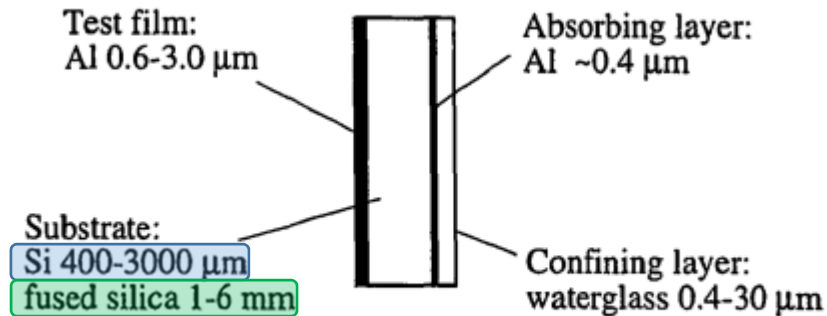


Figure 3.2: Schematic of thin film sample for spallation experiment.

Ex. 1005, pp. 44-45.

As a comparison between the bare silicon substrate and the fused silicon substrate, Wang studied spallation damage patterns formed in the substrates. Ex. 1005, pp. 46-51. Wang explains that damage to the substrate can occur following laser impingement on the absorbing layer, *which is not desired in laser-spallation experiments as this causes the test film to be removed from the substrate prior to any interface failure.* Ex. 1005, p. 47.

a. Silicon Substrate Exhibit Damage Patterns

Experiments on the silicon substrate also showed two distinct damage patterns, a crater formed on the surface and a donut-like damage pattern. Ex. 1005, p. 64. The Wang-Dissertation explains that these damage patterns are typical for silicon substrates and not desired in laser spallation experiments as they result in

causing the test film to come off along with the substrate in one piece without the failure of the desired interface. Ex. 1005, pp. 47, 61.

Damage patterns for aluminum films deposited on 600 μm thick silicon substrates are shown in FIG. 3.3.

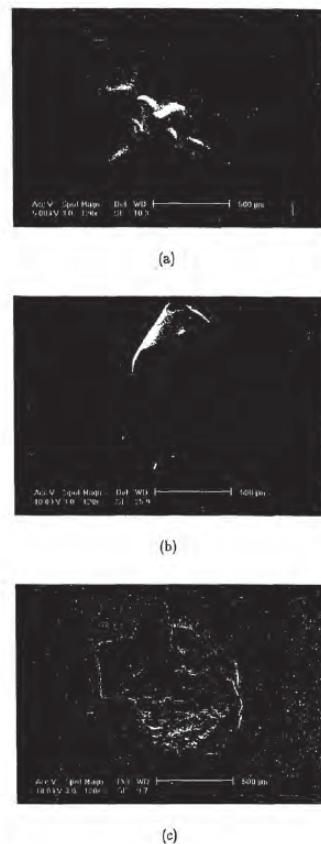


Figure 3.3: Damage observed for 1.0 μm thick aluminum film deposited on 600 μm thick silicon substrate: (a) film lift-off; (b) film spallation, no substrate damage; (c) substrate damage plus film spallation.

As explained in the Wang-Dissertation, in order evaluate the damage in the samples, a Sloan DekTak³ST stylus surface profilometer was used to scan the surface of the damages spot. Ex. 1005, p. 47. Figures 3.4a and 3.4b, reproduced below, show damaged aluminum film on the silicon substrate similar to Figs. 3.3b and 3.3c.

Id. Figures 3.4c and 3.4d show the scanned data in a plotted format. *Id.* The Wang-Dissertation indicates that “the profilometer trace clearly shows the extensive damage caused in the silicon substrate.” Further, Wang explains that damage patterns similar to Fig. 3.3a were not suitable to examination using the profilometer because the lifted film is torn away by the stylus. *Id.*

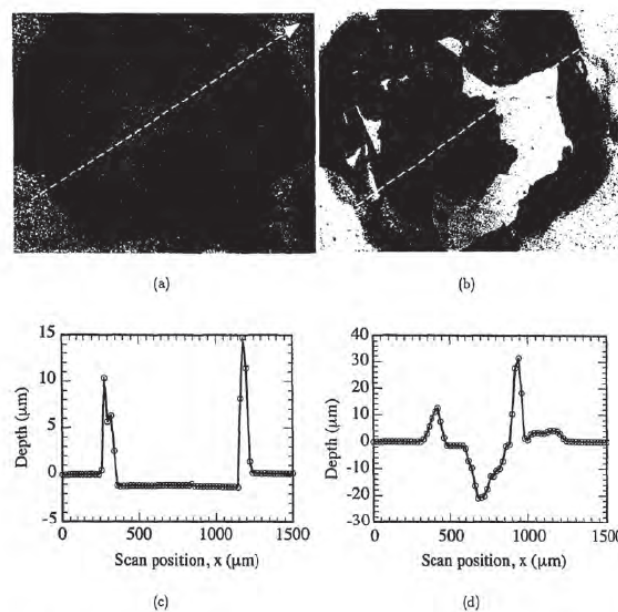


Figure 3.4: Damage observed for 1.2 μm thick aluminum film deposited on 600 μm thick silicon substrate: (a) no substrate damage, only film spallation; (b) substrate damage plus film spallation; (c) height profile corresponding to (a); (d) height profile corresponding to (b).

Fig. 3.4 Showing Damage Patterns and Plots (Ex. 1005, p. 49)

The Wang-Dissertation concludes that because “the primary interests of this project is to measure the interface adhesion between the film and the substrate, substrate damage (Fig. 3.4b) is not desired.” *Id.*

b. Fused Silica Substrates Do Not Exhibit Damage

In contrast to the silicon substrate testing, experiments on silica substrates showed no damage due to their intrinsic strength making them suitable for laser spallation testing. Ex. 1005, pp. 51, 61-61. Testing on the fused silica substrates resulted the in damage patterns shown in Fig. 3.6 reproduced below. These fused silica substrate samples showed no substrate damage.

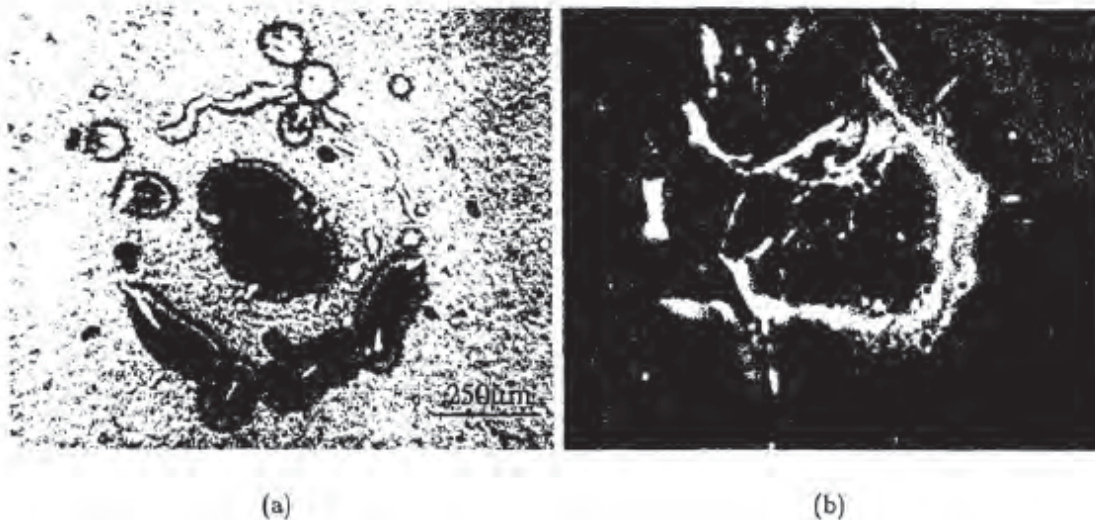


Figure 3.6: Typical damage patterns for a 1.9 μm thick aluminum film deposited on a 3000 μm thick fused-silica substrate sample: (a) lower laser fluence (0.07 J/mm^2); (b) higher laser fluence (0.140 J/mm^2). The diameter of the laser spot is about 1 mm.

Fig. 3.6 Damage Patterns on Fused Silica (Ex. 1005, p. 51)

5. Substrate Stress Attenuation in the Substrate

The Wang Thesis describes that there is strong stress attenuation with respect to substrate thickness. Ex. 1005, p. 61. Investigations show that for the silicon substrate and the fused silica substrate studied at different thicknesses, maximum

substrate stress and maximum interface stress are lower for greater substrate thickness. Ex. 1005, pp. 68-69. Experiments show that maximum interface stress drops with increasing substrate thickness and this impact on maximum interface stress is significantly higher for silicon substrates as compared to fused silica substrates at the same thickness, albeit at substrate thickness less than 3000 μm . For a thickness of 3000 μm , the maximum interface stress for the silicon substrate is 75 as compared to 350 for the fused silica as shown in Tables 3.3 and 3.4 reproduced below. Ex. 1005, p. 69. A nearly 5X drop in interface stress.

Table 3.3: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on silicon samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	400 μm	600 μm	3000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.7	-1.4	-0.55
$\sigma_{\text{int}}^{\text{max}}$	100	90	75

Table 3.4: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on fused silica samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	1500 μm	3000 μm	6000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.2	-0.5	-0.3
$\sigma_{\text{int}}^{\text{max}}$	370	350	50

B. Gupta-1992 (Ex. 1011)

1. Introduction

Gupta-1992 relates to measurement of interface strength of planar interfaces between a substrate and a thin coating. Ex. 1011, Abstract, pp. 141-142. Noting the

problems of interface reaction between a matrix and a fiber may result in premature failure, Gupta-1992 seeks to measure interface strength of sacrificial coatings applied to fiber based materials to permit controlled delamination of the coating from the fiber along the fiber-coating interface to prevent cracks external to the fiber. Ex. 1011, p. 141. By tailoring the strength and toughness properties of the fiber-coating interface, these coatings provide mechanical fuses to decouple undamaged fibers from their damages surroundings to prevent catastrophic failure of the part. Ex. 1011, pp. 141-142. Important to realizing these effects is measurement of the tensile strength and intrinsic delamination toughness of the fiber-coating interface. Ex. 1011, p. 142. Gupta-1992 discusses how the tensile strength of the fiber-coating interface may be measured using a laser spallation technique. Ex. 1011, p. 142.

2. Model for Coating Interface Strength Measurement

As the actual interfaces described above are between a cylindrical fiber and a coating, Dr. Gupta recognized these surfaces are not accessible to measurement using a spallation technique. Ex. 1011, p. 142. Accordingly, the approach used in Gupta-1992 utilized a planar measurement technique using a model substrate and coating combination as shown below in Fig. 1.

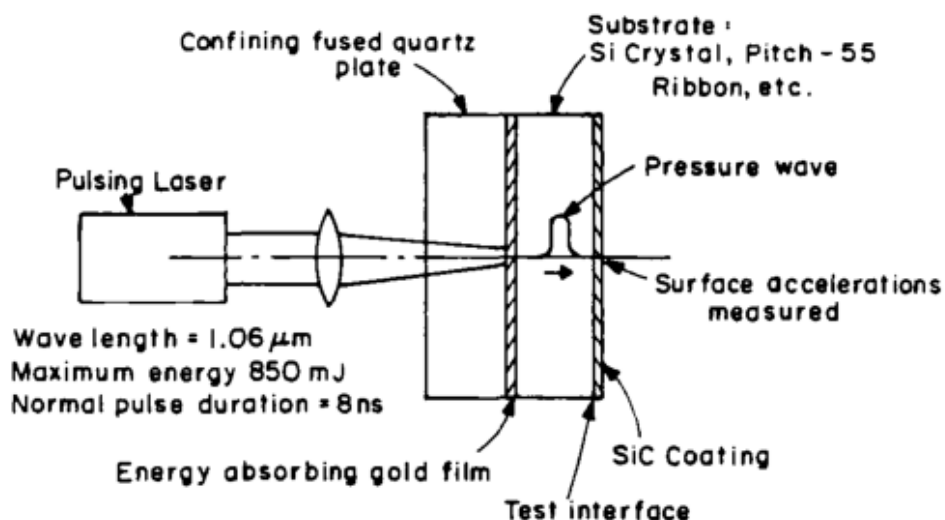
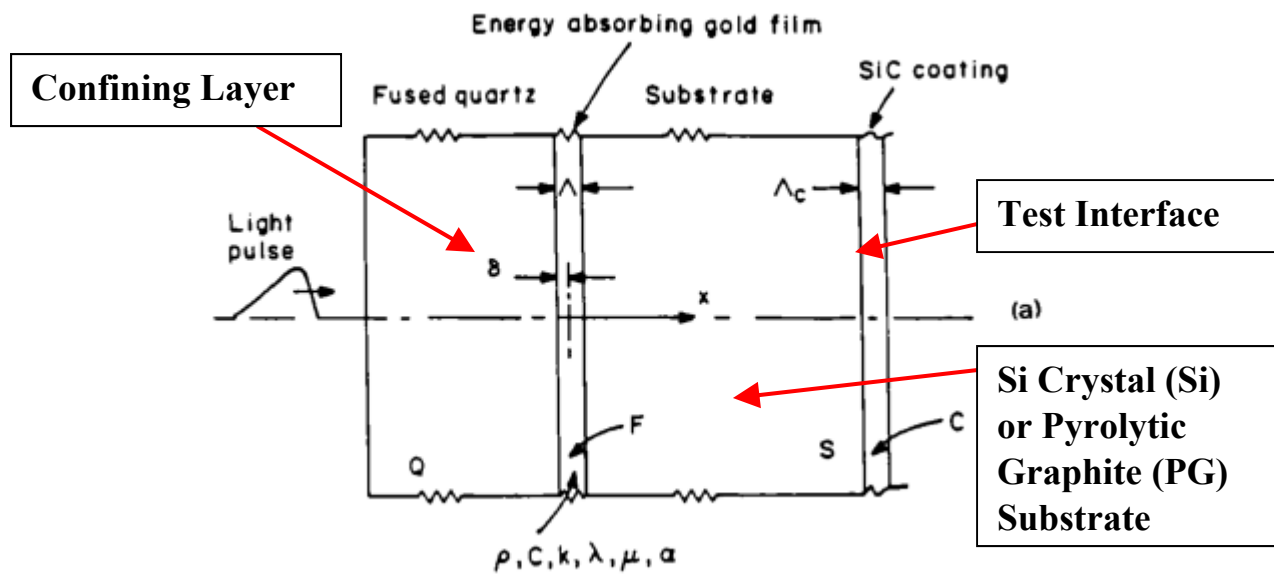


FIG. 1. Schematic view of the laser spallation experiment.

To perform laser spallation, a collimated laser pulse impinges thin energy absorbing film sandwiched between a fused quartz confining plate and a substrate of interest, *e.g.*, Si crystal (Si), Pitch -55 ribbon and pyrolytic graphite (PG), to measure the strength of the corresponding substrate/SiC coating interface. Ex. 1011, pp. 142, 144.

3. Configuration for Si Crystal and Pyrolytic Graphite Substrate Model Testing

Experiments for determining the interface strength were performed under two different structural configurations. In the case of the Si/SiC and PG/SiC interface systems, the experimental configuration was of the type shown in Fig. 2a below:

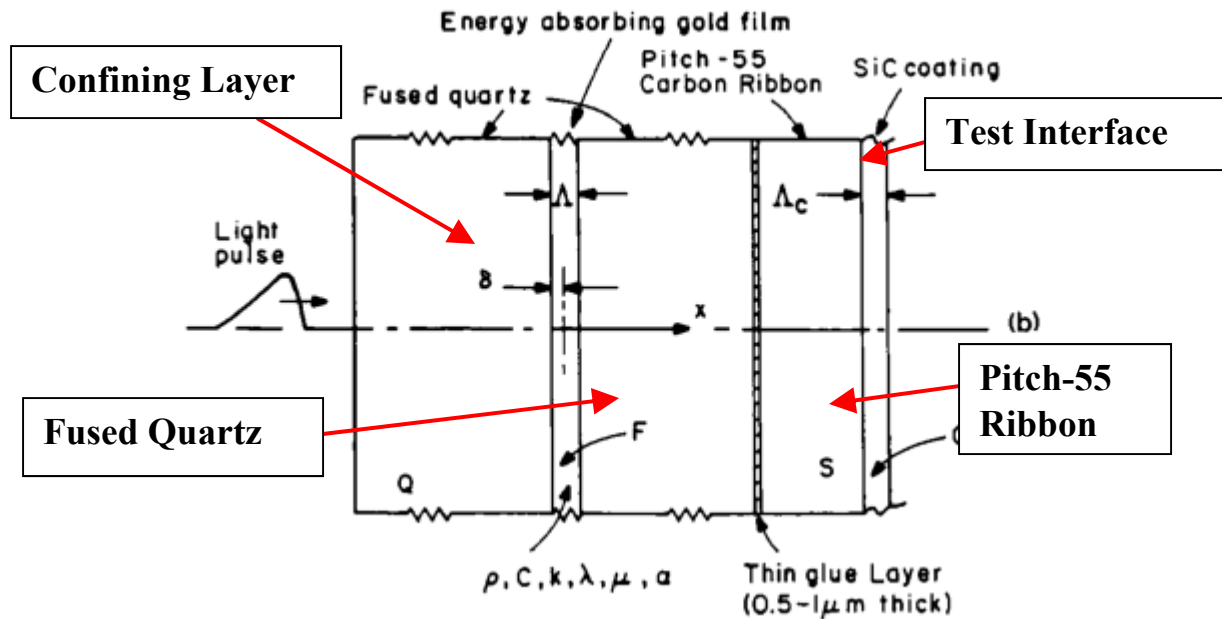


Annotated Fig. 2(a) from Gupta-1992

The substrate of Fig. 2(a) was either a Si crystal (Si) substrate or a pyrolytic graphite (PG) substrate and the test interface was an SiC coating. Ex. 1011, p. 144-145. Experimental results indicated average interface strength for the Si/SiC system at the Si/SiC coating interface ranged from 3.7 to 10.88 GPa depending methods used to deposit the SiC coating. Ex. 1011, p. 175. For the PG/SiC system, the PG/SiC interface strength ranged from 3.68 to 7.48 GPa. No fractures in the Si or PG substrates were realized. Ex. 1011, p. 175.

4. Configuration for Fused Quartz Substrate Model Testing

In a second structural configuration, interface strengths were tested for the Pitch-55 ribbon/SiC interface using the structure of Fig 2(b) as shown below:



Annotated Fig. 2(b) from Gupta-1992

Having a different structure than used in Fig. 2(a), testing of the Pitch-55/SiC coating interface additionally included a fused quartz substrate between the confining layer and the Pitch-55 Carbon Ribbon as shown in Fig. 2(b). Experimental results indicated average interface strength for the fused quartz/Pitch-55/SiC system at the Pitch-55/SiC coating interface ranged from only 0.22 to 0.26 GPa. Ex. 1011, p. 175. Gupta-1992 explains that these results were most likely governed by fractures within the Pitch-55 ribbon itself. *Id.* The interface strength in the range of 0.22 to 0.26 GPa is lower with the use of fused quartz compared with 3.7 to 10.88 GPa for the Si/SiC system.

C. Wang-2002 (Ex. 1011)

1. Wang-2002 Overview

Wang-2002 relates to a method of performing thin-film adhesion measurement using laser-induced stress waves. The structure used in Wang-2002 is the same as the described in the Wang-Dissertation (Ex. 1005) above. Fig. 1 of Wang-2002 describes the general structure of the laser spallation experiment structure. Ex. 1011, pp. 75-76.

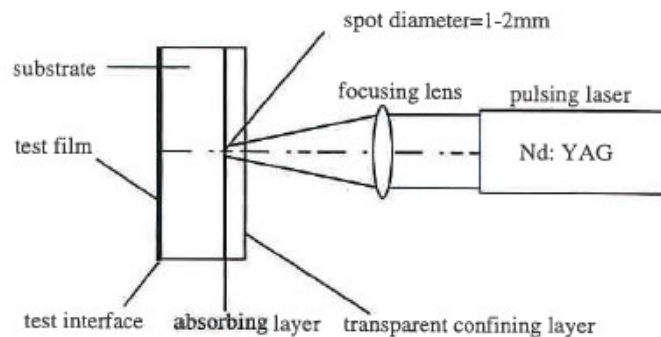


Fig. 1—Schematic of laser spallation technique: absorbing layer thicknesses are typically between 0.4 – 1.0 μm , test film thicknesses are between 0.6 – 3.0 μm and the substrate has a thickness between 400 – 6000 μm

Using a Nd:YAG pulse is incident on a metallic absorbing layer sandwiched between the transparent confining layer and the substrate to induce a longitudinal stress wave that propagates through the substrate toward the film/substrate interface (test interface). Ex. 1011, p. 75. Laser energy impinging the adsorbing layer confined by the substrate and the confining layer induces a compression stress wave

in the substrate directed toward the test film. Ex. 1011, p. 74. The reflection of these compression stress wave from the surface of the test film generates a tensile pulse which may lead to spallation of the test film. Ex. 1011, p. 74. By measuring displacement at the free surface of the test film, the stress history of the interface is determined based on standard wave mechanics and the maximum stress acting on the interface is then calculated. Ex. 1011, p. 75 (Abstract).

2. Testing of Fused Silica as the Substrate

Wang-2002 reported parametric studies comparing the use of silicon and alternatively fused silica as a substrate for testing as shown in Fig. 3 reproduced below. Spallation of thin Al films were investigated as a function of substrate thickness, film thickness and laser energy. Ex. 1016, p. 74 (Abstract). Many tests revealed realized substrate stress damaged the Si substrate and showed the maximum stress at the test interface decreasing with increasing substrate thickness. Ex. 1016, p. 74 (Abstract). In contrast to the Si substrate, the fused silica substrate developed a decompression shock resulting in a high tensile stress at the interface with no substrate damage.

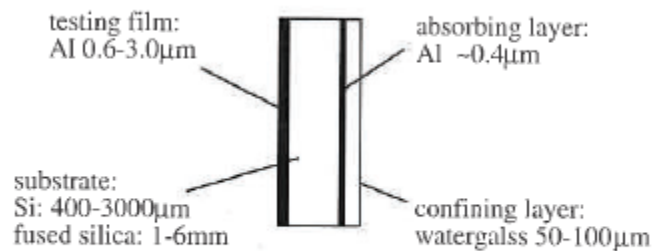


Fig. 3—Schematic of thin film sample for spallation experiment

3. Problems Inhibiting Tensile Stress Formation at the Test Interface and Measuring Adhesion Strength

Wang-2002 identifies the similar problems identified in the Wang-Dissertation (Ex. 1005): (1) attenuation of stress due to the thickness of the substrate; (2) realized tensile stresses at the test interface being lower than the stress acting on the substrate as the free surface of the test film releases some of the stress; and (3) damage to the substrate. Ex. 1016, p. 76-77.

4. Substrate Attenuation

Wang-2002 describes the influence of substrate thickness for both fused silica and Si substrate samples. Ex. 1016, p. 81. Three different thicknesses of fused silica (1531, 3063 and 6125 μ m)(Table 3) and silicon (400, 600 and 3000)(Table 4) were tested to determine the maximum interface stress realized at the coating/substrate interface.

TABLE 3—EFFECT OF SUBSTRATE THICKNESS ON Si
SUBSTRATE SAMPLES

Substrate thickness (μm)	400	600	3000
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	−1.7	−1.4	−0.55
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	100	90	75

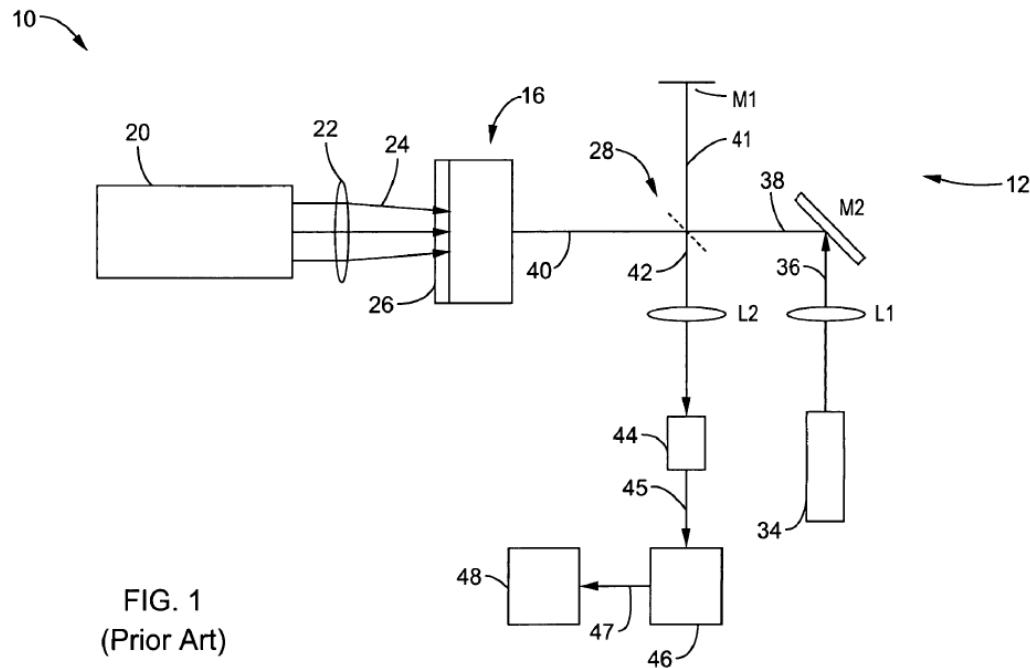
TABLE 4—EFFECT OF SUBSTRATE THICKNESS ON
FUSED SILICA SAMPLES

Substrate thickness (μm)	1531	3063	6125
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	−1.2	−0.5	−0.3
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	370	350	50

III. THE ‘684 PATENT

The ‘684 Patent is a continuation application No. PCT/US2005/007304 filed March 7, 2005, which claims priority from U.S. provisional application No. 60/550,803 filed March 5, 2004. An even earlier actual reduction to practice of the claimed invention is also described in the article title “Glass-modified stress waves for adhesion measurement of ultra thin films for device applications,” authored by Gupta et al. Ex. 2001.

The ‘684 Patent generally relates to an apparatus and method for generating laser spallation of ultra thin films and measuring the interface tensile strength between a substrate and a film using glass modified stress waves. Ex. 1001, 1:48-52; 2:48-57; FIGS. 4, 6 and 8A. In operation, laser spallation has been performed conventionally in the following configuration as shown in FIG. 1 of the ‘684 Patent:



When actuated, the first input laser 20 generates a laser pulse that passes along the first axis to the lens 22, which collimates the laser pulse into a collimated beam 24 incident upon a constraining layer 50. Absorption of the laser pulse by the energy absorbing layer 52 generates a compressive shock wave or pulse directed towards the substrate 56 and the test coating 58 deposited on the substrate 56 front surface. Ex. 1001, 5:50-7:26.

FIG. 3 reproduced below shows the stress wave profiles produced by conventional laser spallation configurations.

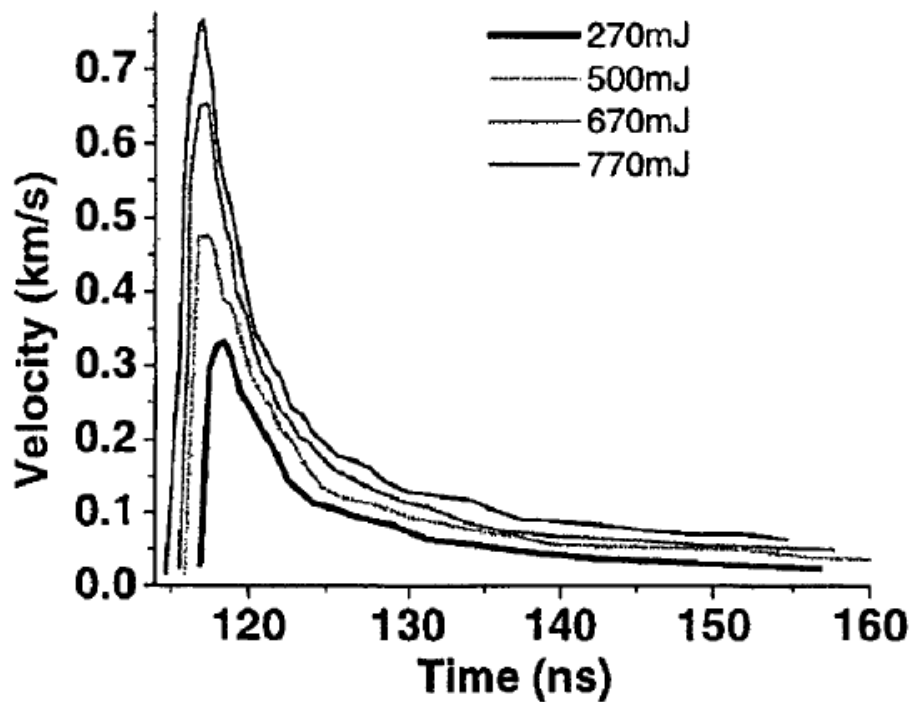


FIG. 3
(Prior Art)

In the present application, FIG. 4 (reproduced below) illustrates an exemplary sample assembly additionally including a glass substrate element 106 as part of the structure between the energy absorbing layer 104 and the silicon substrate 108. Ex. 1001, 8:52-

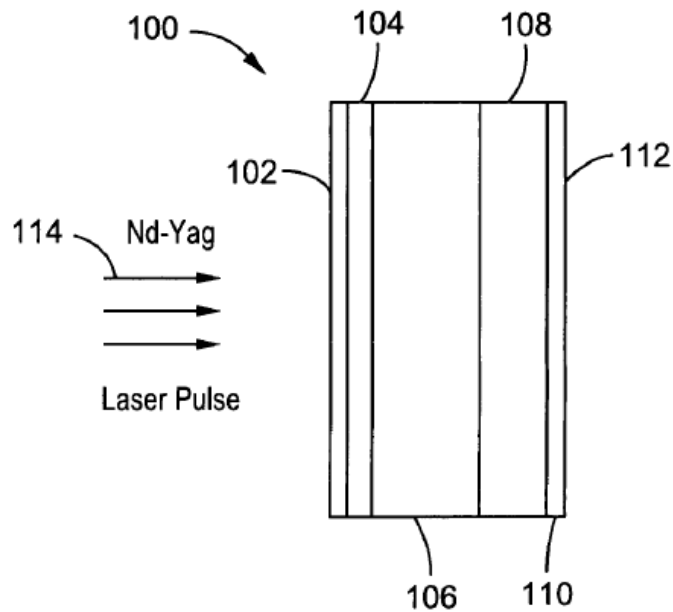


FIG. 4

In testing the of this configuration, the coating-free surface of the Si substrate 108 was bonded using an UV-cured epoxy to a Pyrex, quartz or borosilicate glass layer 106 to allow loading due to glass-modified waves. Table II illustrates the failures observed at the interfaces of interest for the various configurations, with specific tensile strength values for each configuration. Ex. 10:10-22. The comparison between sample no. 1 and sample no. 2 shows successful the successful interface failure when using borosilicate glass as the glass element.

TABLE II

Interface Tensile Strengths Measured Using Glass-Modified Stress Waves.				
Sample No.	Multilayer Configuration	Peak Interface Tensile Stress (MPa)	Peak Tensile Stress in Substrate (MPa)	Failure Locus
1.	Cu(1400 nm)/TiN(70 nm)/Si	451	5340	Silicon failure
2.	Cu (1400 nm)/TiN(70 nm)/Si Borosilicate glass	2620 ± 2120		Cu/TiN interface
3.	Mo[400 nm]/Borosilicate glass	577 ± 47	2624	Mo/Borosilicate glass interface
4.	Mo[1000 nm]/Borosilicate glass	245 ± 26	1050	Borosilicate glass Failure
5.	Mo[400 nm]/Borosilicate glass	577 ± 33	2624	Mo/Borosilicate glass interface
6.	Mo[400 nm]/Pyrex	135 ± 11	1125	Mo/Pyrex interface
7.	Mo[400 nm]/Quartz	99 ± 8	1110	Mo/Quartz interface
8.	Mo[1000 nm]/Borosilicate glass	233 ± 24	980	Borosilicate glass failure
9.	Mo[1000 nm]/Pyrex	53 ± 6	470	Borosilicate glass failure
10.	Mo[1000 nm]/Quartz	48 ± 4	460	Borosilicate glass failure
11.	TiN[320 nm]/Borosilicate Glass	164 ± 11		TiN/Borosilicate glass interface
12.	Ni[350 nm]/Borosilicate Glass	120 ± 13		Ni/Borosilicate glass interface
13.	Cr[400 nm]/Borosilicate glass	197 ± 16	2100	Cr/Borosilicate glass interface
14.	Mo[1000 nm]/Cr[400 nm]/Borosilicate glass	210 ± 19	980	Cr/Borosilicate glass interface

IV. CLAIM CONSTRUCTION

Boeing contends no terms need be construed because the prior art discloses a limitations under the plain meaning as well as any plausible construction. Further, Boeing contends that “the first named inventor/PO’s exclusive licensee has put forth a broad construction for ‘glass element’” citing to Ex. 1033 at ¶10. Pet., p. 4 (footnote 1). Those portions of Ex. 1033 contend that the ’644 Patent uses a “glass element” to shape the stress wave which in turn results in the generation of a much higher

tensile force at the interface. Ex. 1033 at ¶10. Boeing does not disagree with this construction in its Petition.

V. A PERSON OF ORDINARY SKILL IN THE ART

For the purposes of this Preliminary Response, PO generally agrees with the Petition's definition of a POSITA.

VI. THE BOARD SHOULD DENY INSTITUTION BECAUSE THE PETITION FAILS TO ESTABLISH A LIKELIHOOD OF SUCCESS FOR ALL GROUNDS ASSERTED FOR INDEPENDENT CLAIMS 1 AND 13

A. Ground 1 – Anticipation By Wang-Dissertation

1. The Petition Fails to Establish Public Availability of the Wang-Dissertation

The Petition fails to establish a likelihood of success in showing anticipation of claims 1 and 13 because it fails to establish a date of public availability of the Wang-Dissertation. Oddly, the date the Petition asserted as the date of public availability is March 4, 2003, just one day longer than a year prior to the filing date the Provisional Application No. 60/550,803 from which the '684 Patent claims priority. Asserting this date without any proper documentation or real evidence undermines the contention. Otherwise, the deficiencies of the Petition's contentions are outlined below.

“Whether a . . . document qualifies as a ‘printed publication’ under § 102 is a legal conclusion based on underlying factual determinations.” *SRI Int’l, Inc. v. Internet Sec. Sys., Inc.*, 511 F.3d 1186, 10 1192 (Fed. Cir. 2008) (citation omitted). “‘Public accessibility’ has been called the *touchstone* in determining whether a reference constitutes a ‘printed publication’ bar under 35 U.S.C. § 102.” *Id.* at 1194 (citation omitted). A reference is publicly accessible upon a satisfactory showing that it has been disseminated or otherwise made available to the extent that persons interested and ordinarily skilled in the subject matter or art exercising reasonable diligence, can locate it. *Bruckelmyer v. Ground Heaters, Inc.*, 445 F.3d 1374, 1378 (Fed. Cir. 2006); *see also In re Cronyn*, 890 F.2d 1158, 1160 (Fed. Cir. 1989) (“The statutory phrase ‘printed publication’ has been interpreted to mean that before the critical date the reference must have been sufficiently accessible to the public interested in the art; dissemination and public accessibility are the keys to the legal determination whether a prior art reference was ‘published.’”) (quoting *Constant v. Advanced Micro-Devices, Inc.*, 848 F.2d 1560, 1568 (Fed. Cir. 1988)).

In an IPR, the petitioner bears the burden of establishing by a preponderance of the evidence that a particular document is a printed publication. *Medtronic, Inc. v. Barry*, 891 F.3d 1368, 1380 (Fed. Cir. 2018).

The “printed publication” analysis under § 102 “involves a case-by case inquiry into the facts and circumstances surrounding the reference’s disclosure to

members of the public.” *In re Klopfenstein*, 380 F.3d 1345, 1350 (Fed. Cir. 2004) (citations omitted).

Generally, for a document stored in an electronic database, a keyword would be needed to find the document. Chronological or alphabetical indexing is not sufficient, as a member of the interested public would be unlikely to locate the document of interest. To qualify as a printed publication, a reference “must have been sufficiently accessible to the public interested in the art.” *In re Cronyn*, 890 F.2d 1158, 1160-61 (Fed. Cir. 1989) (reasoning that references were not publicly accessible, despite the use of indexing, because the references were indexed only by title and author's name, rather than by subject).

In addition, for a document to be publically available on a particular date, the document would need to have been able to be actually received by a member of the interested public by that date. Boeing failed to meet this standard. “[T]he one who wishes to characterize the information, in whatever form it may be, as a “printed publication” ... should produce sufficient proof of its dissemination or that it has otherwise been available and accessible to persons concerned with the art to which the document relates and thus most likely to avail themselves of its contents.” *In re Wyer*, 655 F.2d 221, 227 (C.C.P.I. 1981) (citations omitted).

A declaration is sufficient to show public availability of a reference only if it can be corroborated with other evidence. “[C]orroboration is required of any witness

whose testimony alone is asserted to invalidate a patent, regardless of his or her level of interest.” *Finnigan Corp. v. Int’l Trade Comm’n*, 180 F.3d 1354, 1369 (Fed. Cir. 1999). Corroborating evidence may include documentary or testimonial evidence. *See TransWeb, LLC v. 3M Innovative Props. Co.*, 812 F.3d 1295, 1301 (Fed. Cir. 2016).

In this case, one of the declarations relied upon by Boeing in its effort to establish public availability of the Wang-Dissertation is Exhibit 1035, the Declaration of Ms. LaTonya Morris (“Morris Declaration”). The Morris Declaration is not credible or legally sufficient for various reasons.

First, the declaration is presented by an employee of ProQuest who has worked for the company only since 2018, but the employee nevertheless attempts to establish that the Wang-Dissertation was publically available in 2003. Accordingly, the declaration is not based on first-hand evidence, as it seeks to make assertions about the history of a ProQuest database during a period that was 15 years prior to the time when the deponent began working at ProQuest. In the declaration, the deponent does not explain how she knew about the capabilities of the ProQuest database from this period, so the basis for and accuracy of her assertions is left in doubt.

Second, the Morris Declaration asserts that there is a ProQuest records database which includes a citation that has author name, title, and date. Exhibit 1035

at ¶ 3. The declaration provides a table with the purported information for the Wang-Dissertation, but does not provide an actual printout from the database. *Id.* at ¶ 8. The Morris Declaration further asserts, without evidence, that this database has an “Abstract.” *Id.* at ¶ 11. A copy of the purported “Abstract” is not provided, so that statement is uncorroborated.

The Morris Declaration alleges that a person would be able to search a first ProQuest database for the Abstract and arrive at the Wang-Dissertation as a potential reference. *Id.* at ¶¶ 8-12. The declaration then asserts that, once one identifies Wang-Dissertation, there is a second ProQuest database from which you can purchase the actual thesis. *Id.* at ¶¶ 13-15.

The pre-AIA version of 35 U.S.C. § 102(b) states, in relevant part: “A person shall be entitled to a patent unless ... the invention was patented or described in a printed publication in this or a foreign country or in public use or on sale in this country, more than one year prior to the date of the application for patent in the United States.” Boeing has alleged that the earliest effective filing date of the ’684 Patent is March 5, 2004. IPR Petition at p. 4. Boeing further asserts that the Wang-Dissertation published on March 4, 2003. *Id.*; Exhibit 1035 at ¶ 9. Accordingly, Boeing has asserted that the Wang-Dissertation published one year and one day prior to the earliest effective filing date of the ’684 Patent. This means that to qualify as

a prior art reference under pre-AIA Section 102(b), the Wang-Dissertation needs to have been accessible on March 4, 2003.

In summary, in order for an interested person to avail herself of the Wang-Dissertation as a Section 102(b) prior art reference, she would have to use the right keywords when searching the first database to arrive at the reference and then go to the second database and order the dissertation and get it delivered on the same day – March 4, 2003. However, there are multiple problems with such a scenario, and the probability of it occurring are very low if not impossible.

As in initial matter, Boeing has not shown that the Abstract of the Wang-Dissertation is actually available for search. *See* Exhibit 1006, Exhibit 1036. Even if the Abstract were available, there are no keywords provided. In 2003, at the time that Boeing alleges that the Wang-Dissertation was first available, a full-text search of the Abstract could not be expected. In fact, when documents were uploaded to such databases at the time, it would have been a routine practice to ask for keywords.

Even if keyword search could have been done, the words that are in the claim that are important to this invention or their equivalents are not in the Abstract. For example, there is no mention of “glass substrate” or any modification of stress wave, which are the key improvements over prior art.

Even if the abstract of the Wang-Dissertation were somehow identified in the first ProQuest abstract database, one would have to order the actual dissertation

using the second ProQuest database. In 2003, while one could order using credit cards, it was not processed instantaneously as today. And there is no indication given by Boeing that same-day delivery was even possible at the time. Accordingly, it was highly unlikely, and perhaps impossible, for the Wang-Dissertation to be located on the date it was first published (March 4, 2003) and then delivered on the same date to qualify as 102(b) reference. Boeing attempted to prove no other date of alleged public availability. If the Wang-Dissertation is only available under 102(a), an actual reduction to practice of the claimed invention is also described in the article title “Glass-modified stress waves for adhesion measurement of ultra thin films for device applications,” authored by Gupta et al. Ex. 2001 is available to support an earlier date.

2. The Petition’s Anticipation Contention

The Petition alleges that the Wang-Dissertation anticipates all elements of claims 1-24 of the ‘684 Patent. In particular, the Petition alleges the Wang-Dissertation discloses all features of claims. As set forth below, Patent Owner disagrees that the Wang-Dissertation anticipates the features of claims 1 and 13 identified below:

Claim 1

“a substrate having a thickness, said thickness defined by a first side and a second side in a first axis;”

“a glass element disposed on the second side of the substrate and axially spaced along the first axis;

wherein the glass element is configured to propagate and modify a stress wave to the coating/substrate interface to generate a tensile stress between the substrate and the coating.”

Claim 13

“a substrate, the substrate having a first side and a second side transversely disposed in a first axis”

“positioning a glass element along the first axis on the second side of the substrate;”

“propagating and modifying a stress wave through the glass element to the coating/substrate interface to generate a tensile force between the substrate and the coating;”

Only two different substrate patterns were studied by Junlan Wang: (1) fused silica and (2) silicon. Absent is any structural detail described that uses fused silica as a “glass element” to modify the stress wave in combination with a “silicon substrate/coating” interface. The fused silica substrate and the silicon substrate were tested independently of each other, never in combination.

As support in the Wang-Dissertation for using a “glass element” in combination with another “substrate” as recited in claims 1 and 13, Boeing cites to a portion in the thesis titled “5.2 Future Work”:

By inserting an extra layer of the desired substrate material between the fused silica substrate material and the thin film, the current technique can be applied to test a variety of substrates and films, especially those of interest to the microelectronics industry, such as polyamide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates.

Ex. 1005, p. 124.

These combinations were never tested or shown within the Wang-Dissertation. Moreover, Wang-Dissertation lays out in detail the problems associated with stress attenuation and substrate damage associated with its silicon substrate, yet neither the Petition nor the Wang Declaration address how any purported combination of the two different substrates, fused silica and silicon, would alleviate or overcome these problems.

3. The Wang-Dissertation Fails to Anticipate the Independent Claims

The Wang-Dissertation fails to anticipate independent claims 1 and 13 as the work of Junlan Wang only describes embodiments that use a single substrate – either fused silica or silicon. No combinations of different substrates in combination with a fused silica substrate are described or enabled. As these combinations were never

enabled, Boeing's reliance on statements of "Future Work" does not teach one of skill how to make and use the "fused silica" in combination with another "substrate" to perform interfacial tests as alleged in the Petition. As will be shown below, the Wang-Dissertation itself provides ample evidence that this "Future Work" is not enabled.

Junlan Wang performed experiments on either a bare silicon substrate or a fused silica substrate having the general structure shown in FIG. 3.2 below. These were never tested in combination.

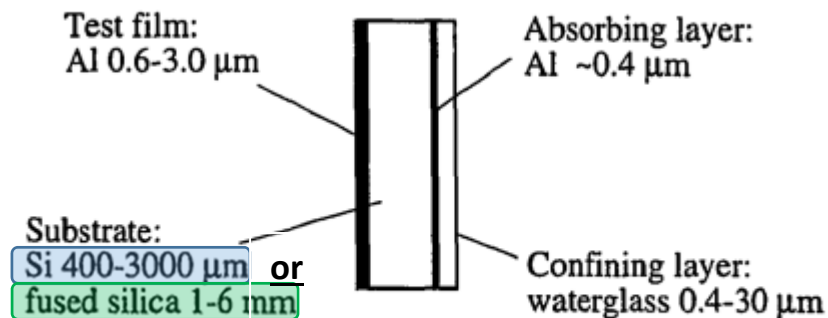


Figure 3.2: Schematic of thin film sample for spallation experiment.

Ex. 1005, pp. 44-45 (annotated).

Claims 1 and 13 both require a "glass element" disposed on a "second side of the substrate" with the "coating applied to the first side of the substrate." That is, the claims require a substrate having glass element on one side and a coating on the

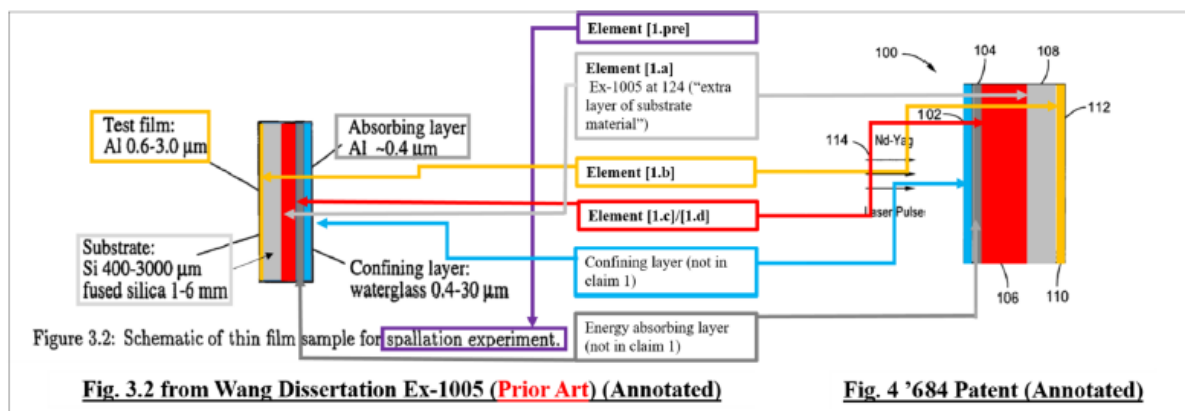
other side. No such configuration is shown by Wang. As support for these features, Boeing cites to a portion in the thesis titled “5.2 Future Work”:

By inserting an extra layer of the desired substrate material between the fused silica substrate material and the thin film, the current technique can be applied to test a variety of substrates and films, especially those of interest to the microelectronics industry, such as polyamide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates.

Ex. 1005, p. 124.

While the Wang-Dissertation fails to describe any specific structure, any tests, or any experiments for this configuration, Boeing provides the following annotated figure modifying Fig. 3.2 as follows:

Figure 3.2 (annotated) shows how Wang anticipates Claim 1:



As explained below, in view of the clear problems of substrate damage and attenuation in a silicon substrate as compared to the fused silica (material with non-

linear properties and intrinsically strong), the Wang-Dissertation fails to enable one of skill in the art to make and use a fused silica “glass element” and an additional “substrate” combination as annotated by Boeing above.

4. The Wang-Dissertation Fails to Enable a Glass Element and Substrate Coupled Structure

The burden to show the prior art is non-enabled lies with the challenger. *In re Kumar*, 418 F.3d 1361, 1368 (Fed. Cir. 2005). *Kumar* explains that when the prior art “appears on its face, to be capable of producing the claimed composition” this burden may be “met by presenting sufficient reason or authority or evidence, on the facts of the case, to show the prior art method would not produce or would not be expected to produce the claimed subject matter.” See also *Beckman Instruments, Inc. v. LKB Produkter AB*, 892 F. 2d 1547, 1550 (1989) (for a prior art reference to be enabling, it must provide a description sufficient to teach a person of ordinary skill in the art how to make and use the apparatus or process). Boeing’s reliance on statements of “Future Work” does not teach one of skill how to make and use the “fused silica” in combination with another substrate to perform interfacial tests, nor does it provide a reasonable expectation of success.

As Petitioner explains below, multiple teachings from the alleged prior art illuminate substantial problems related to **stress attenuation** in both the fused silica

and silicon substrate used in isolation. In the case of the silicon substrate, the Wang-Dissertation describes **substrate damage** as being typical. In view of the known problems of substrate damage and attenuation in a silicon substrate, the Wang-Dissertation fails to enable one of skill in the art to make and use a “glass element” and an additional “substrate” combination. Rather, the Wang-Dissertation merely shows one of skill in the art how to make and use a “fused silica” substrate to perform interfacial adhesion measurements by being bonded directly to a test film due to its nonlinear properties that create “rarefaction shock” to provide high interface stress, and due to survive damage due to its intrinsic strength.

a. Significant Problems in Tested Silicon Substrates as Compared to Fused Silica Substrates Support Non-Enablement

In the experiments covered in the Wang-Dissertation, only fused silica showed successful results, i.e., high interface stress and no substrate damage. In the case of fused silica as the substrate, the maximum interface **stresses realized at the coating/substrate interface were high as desired** – 350 to 370 MPa at thicknesses of 3000 μm and below. Ex. 1005, p. 69. The **fused silica substrate was not damaged** by the tensile stress. Ex. 1005, p. 51, 81 and 120. As explained in the Wang-Dissertation, because the fused silica is intrinsically strong, no substrate damage occurs before interfacial failure. *Id.* at p. 81.

In contrast, when using silicon as the substrate, tests **revealed substrate damage and high stress wave attenuation** - only realizing a stress of 75 MPa at a thickness of 3000 μm . Ex. 1005, p. 69. Additionally, the Wang-Dissertation observed that maximum substrate stress and maximum interface stress become lower for greater substrate thicknesses. Substrate damage is also problematic as noted by Junlan Wang:

The profilometer trace clearly shows the extensive damage caused in the silicon substrate. Damage patterns similar to Fig. 3.3a are not suitable for examination with the profilometer because the lifted film tends to be torn and pushed away by the stylus. Since the primary interest of this project is to measure the interface adhesion between film and substrate, substrate damage (Fig. 3.4b) is not desired.

Ex. 1005, p. 47.

**b. Evidence in the Wang-Dissertation Logically Raises
Concerns Regarding Substrate Damage When Coupled with
Fused Silica**

Boeing posits that the Wang-Dissertation teaches a “fused silica” glass element to be used with one of polyamide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates. Ex. 1005, p. 124. The Wang-Dissertation does not provide any support for how these materials would survive substrate damage in view of the increased tensile stress induced by the fused silica. The Wang-Dissertation describes “damage patterns are typical for silicon substrates”

and these patterns “are not desired in laser-spallation experiments.” Ex. 1005, p. 47. Substrate damage is clearly a concern. With increasing tensile stress by virtue of the additional use of fused silica with a substrate, substrate damage would be more likely, not less likely to occur. Petitioner failed to address substrate damage when relying on the Wang-Dissertation’s “Future Work” description. Considering the wealth of information provided within the Wang-Dissertation, a reasonable expectation of making the fused silica/additional substrate combination is absent. The Petition and the Wang Declaration conveniently ignore this damage issue and only nakedly assert these features are disclosed.

**c. Silicon Substrates Exhibit Damage at Relatively Low
Tensile Stresses Well Below Those Achieved with Fused Silica as
the Substrate**

In the case of silicon substrates, Junlan Wang performed quantitative measurements of displacement, substrate stress and interface stress. Ex. 1005, p. 62. Silicon substrates at a thickness of 600 μm were tested and substrate stress values were plotted in FIG. 3.14(c):

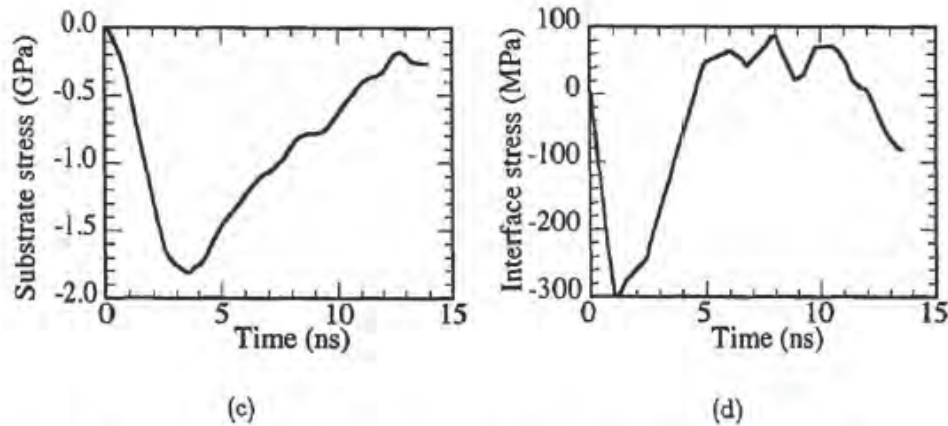


Figure 3.14: A typical data processing cycle for a 1.0 μm aluminum film deposited on a 600 μm thick silicon substrate (laser fluence of 0.140 J/mm²): (a) interferometric fringe pattern; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile.

The maximum interface tensile stress reached 60 MPa. Yet, as noted by the Wang-Dissertation, silicon substrate damage is typical. Ex. 1005, pp. 47 and 62.

d. Fused Silica produces Much Higher Tensile Stresses

Further Inducing Damage in a Coupled Substrate

Junlan Wang also performed quantitative measurements of displacement, substrate stress and interface stress in both the fused silica and silicon substrates. Ex. 1005, pp. 62-66. Effects of substrate thickness on these effects were also tested. *Id.* at pp. 68-69. As shown in Tables 3.3 and 3.4, for a 3000 μm thick substrate, the maximum substrate stresses were similar at -0.55 for silicon and -0.5 GPA for fused silica, but maximum interface stresses were much lower in the silicon substrate at 75 MPa as compared to 350 MPa for the fused silica substrate.

Table 3.3: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on silicon samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	400 μm	600 μm	3000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.7	-1.4	-0.55
$\sigma_{\text{int}}^{\text{max}}$	100	90	75

Table 3.4: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on fused silica samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	1500 μm	3000 μm	6000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.2	-0.5	-0.3
$\sigma_{\text{int}}^{\text{max}}$	370	350	50

These fused silica substrates were tested at 3000 μm thickness and the results were plotted in Fig. 3.15. These tests showed significant differences due to the nonlinear properties of the fused silica, which results in substantial tensile stresses. Notably, the laser fluence used in both the silicon substrate and the fused silica substrate tests were the same at 0.14 J/mm². However, the interface stress realized when using the fused silica substrate was an order of magnitude higher at about 0.6 GPa (600 MPa) as shown in Fig. 3.15(c) reproduced below:

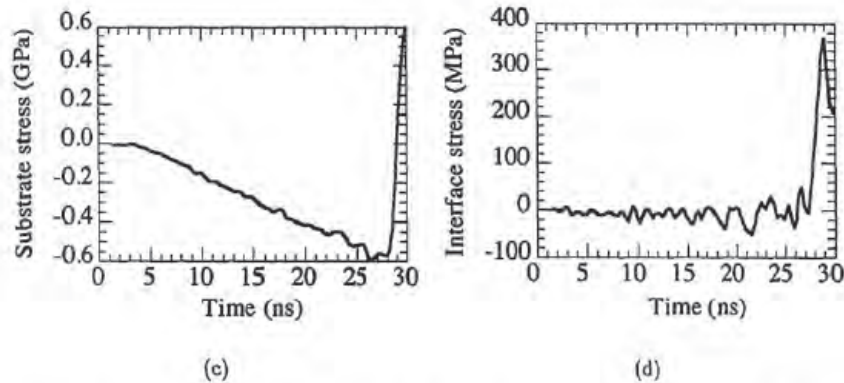


Figure 3.15: Typical data profile for a 1.0 μm thick aluminum film deposited on a 3000 μm thick fused silica substrate (laser fluence of 0.140 J/mm²): (a) interferometric fringe pattern, arrow points to turning point; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile.

As the Wang-Dissertation reports that damage is typical in silicon substrates, it becomes apparent that using a fused silica substrate in combination with a substrate would also lead to higher tensile stresses and would lead one of skill to expect more, not less, substrate damage in such a coupled system. Again, as noted in the Wang-Dissertation, silicon substrate damage is typical and not desired as the primary interest is to measure interface adhesion. Ex. 1005, p. 47.

e. Substrate Attenuation Significantly Impacts Test Interface Stress

The Wang-Dissertation provides clear results using only a fused silica substrate bonded directly to a test film. No insights or evidence are provided to enable one of skill in the art to modify this structure to add an additional substrate,

i.e., polyamide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates.

As experiments show, the significant nonlinear properties of the fused silica will “cause the compressive stress wave generated by the pulsing laser to evolve a decompression shock.” However, as shown in Table 3, substrate thickness substantially impacts the maximum interface stress:

Table 3.3: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on silicon samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	400 μm	600 μm	3000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.7	-1.4	-0.55
$\sigma_{\text{int}}^{\text{max}}$	100	90	75

Table 3.4: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on fused silica samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	1500 μm	3000 μm	6000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.2	-0.5	-0.3
$\sigma_{\text{int}}^{\text{max}}$	370	350	50

As is evident from these results, the silicon substrate thickness attenuates stress and only achieves much lower interface stresses. At the same tested conditions for a 3000 μm thick substrate, while maximum substrate stresses were comparable (-0.55 MPa versus -0.5 MPa), maximum interface stresses were much lower after the compression wave is passed through the silicon substrate.

While the Wang-Dissertation enables one of skill in the art to generate adequate tensile stresses at the substrate/test film interface of 350-370 MPa when using fused silica, the clear evidence of stress wave attenuation show significant decreases in tensile stress when the same laser energy is applied to a silicon substrate. This impact is nearly a 5X drop at 75MPa after the compression wave travels through the silicon substrate. In view of this evidence, absent is any teaching or disclosure in the Wang-Dissertation to enable one of skill in the art to reach the necessary tensile stress of 350-370 MPa reached in fused silica by further transferring the same energy through a subsequent substrate as outlined in the “Future Work” section of the Wang-Dissertation.

It is also worthwhile to note that the Wang-Dissertation describes that “significant attenuation in the peak stress occurs as the stress wave travels through longer distances.” Ex. 1005, p. 38. Even more compelling is that the Wang-Dissertation describes that this “attenuation associated with the shock wave is even greater than that associated with geometric attenuation.” *Id.* Absent is any discussion of how shock wave attenuation in fused silica can be minimized when adding an additional substrate between the fused silica and the test film. The clear evidence in the Wang-Dissertation belies any reasonable expectation of success – and is not addressed in either the Petition or the Wang Declaration.

f. Boeing’s Expert was Informed on Enablement But Failed to Provide Any Support or Evidence Showing the Future Work was Enabled

The declaration of Junlan Wang, Ph.D. (“Wang Declaration”) clarifies that she was informed that “for a reference to be anticipatory, the reference must contain an enabling disclosure” and “that merely naming or describing the subject matter is insufficient if it cannot be produced without undue experimentation.” Ex. 1003, ¶[28]. Further, Junlan Wang was also informed that “a reference will teach away if it suggests that the line of development flowing from the reference’s disclosure is unlikely to be productive of the result sought by the applicant.”

Despite this, the Wang Declaration fails to consider or provide any evidence to counter the clear problems described in the Wang-Dissertation relating to substrate damage typical in the silicon substrate as well as the stress attenuation impact on interface stress. Rather, the Wang Declaration merely provides a conclusory statement from Junlan Wang:

My dissertation discloses that fused silica is an ideal substrate material for launching high-amplitude shock waves (its negative nonlinear elasticity being critical for the development of a decompression shock). Ex-1005, 124. My dissertation further discloses inserting an extra layer of desired substrate material between the fused silica and the thin film to test a variety of substrates and thin films. Ex-1005, 124. A POSITA would have understood in this configuration disclosed in my dissertation, the fused silica would

be a glass element disposed on the second side of the inserted layer of the desired substrate material, e.g., silicon, and axially spaced along the first axis, as show in the figures. Ex-1005, 124, Fig. 2.1, Fig. 3.2.

Ex. 1003, ¶[103].

Junlan Wang ignores the clear problems of substrate damage and substrate attenuation that either make these configurations unworkable for measuring interfacial stress (damage) or unable to provide adequate interfacial stress at the test film interface (attenuation). The declaration fails to address any of these issues and merely provides conclusory statements. *Xerox Corp. v. Bytemark, Inc.*, IPR2022-00624, Paper 9. p. 15 (Aug. 24, 2022) (precedential decision) (A declaration “that merely repeats, *verbatim*, the conclusory assertion for which it is offered to support” is entitled little weight). Accordingly, Patent Owner submits that neither the Petition, the Wang-Dissertation or the Wang declaration support enablement of the Wang-Dissertation.

**5. Conclusion - The Wang Dissertation Fails to Enable One of Skill
in the Art to Make and Use a Fused Silica and Additional
Substrate Combination**

Because the Wang-Dissertation only studied and tested the fused silica and silicon substrates in isolation, in view of the detailed problems of stress attenuation and substrate damage, this reference fails to describe or explain how a substrate

without nonlinear properties could survive the increased tensile stresses produced by the fused silica. The Wang-Dissertation provides strong evidence of stress attenuation in the silicon substrate where a substrate compressive stress of -0.55 GPa only translates to an interface stress of 75 MPa, as compared to the fused silica with values of -0.5 GPa and 350 MPa respectively. Ex. 1005, Tables 3.3 and 3.4. Expectations from this experimental evidence belies using a substrate between the fused silica and the test film – the clear evidence of record shows substrate attenuation is significant and effectively reduces interface stress. Accordingly, the passing statement of “Future Work” relied on in the Petition is just that, work to be done to determine how using an additional substrate, i.e., polyamide, copper (Cu), aluminum (aluminum) and polysilicon on single-crystal silicon substrates. There is no evidence or discussion in the Wang-Dissertation providing insights as to how a “single-crystal silicon substrate” could be utilized with fused silica while avoiding the problems of substrate damage and substrate attenuation – clear problems detailed within the dissertation itself. As noted above, the Wang Declaration fails to address any of these issues and merely provides conclusory statements. Consequently, the Wang-Dissertation fails to enable such a combination.

B. Ground 4 – Anticipation by Gupta-1992

Ground 4 fails to anticipate the following elements of independent claims 1 and 13:

Claim 1

“a glass element disposed on the second side of the substrate and axially spaced along the first axis;

wherein the glass element is configured to propagate and modify a stress wave to the coating/substrate interface to generate a tensile stress between the substrate and the coating.”

Claim 13

“positioning a glass element along the first axis on the second side of the substrate;”

“propagating and modifying a stress wave through the glass element to the coating/substrate interface to generate a tensile force between the substrate and the coating;”

The Petition alleges that Gupta-1992 teaches the use of fused quartz glass in the same configuration as shown in the ‘684 Patent. Notably absent from the Petition or the Wang Declaration is any evidence to support this “fused quartz” is configured to “propagate and modify a stress wave to the coating/substrate interface” as required by independent claims 1 and 13. Pet. , p. 48-50. To the contrary, the Petition provides no supporting evidence that the fused quartz used in Gupta-1992 is configured to modify a stress wave. In fact, Boeing’s expert Dr. Wang expressly concedes that its “intended function and the scientific mechanisms were not

explicitly discussed in the paper”. Ex. 1003, p. 79. Further, without a scintilla of evidence or logical reasoning, Dr. Wang seeks to cure this deficiency by merely stating these properties would be “anticipated based on the known knowledge of shock development in this material (Ex-1021) in this specific configuration.” *Id.* But, Exhibit 1021 by Barker and Hollenbach fails to support this contention. Rather, Exhibit 1021 describes shock development in high-purity fused-silica, not fused quartz. Ex. 1021, p. 4209 (2nd column, first full paragraph). Absent is any support for Boeing’s and Dr. Wang’s conflating use of fused silica with fused quartz. Most compelling, however, Dr. Wang admits in the Wang-Dissertation that “[a]lthough shock development in fused silica was demonstrated in the 1970s by Barker and Hallenbach using a gas-gun facility, the usefulness of such shocks in applications to laser spallation has not been reported.” Ex. 1005, p. 81. Yet, the Wang-Dissertation cites to Gupta-1992 and is thoroughly discussed and cited. *See* Ex. 1005, pp. 4, 8, 9, 22, 40 and 42. Clearly, the post hoc conclusions in the Petition and Wang Declaration belie Boeing’s own supporting documents as well as Dr. Wang’s prior written statements that fused silica has never been tested with laser spallation.

Otherwise, Gupta-1992 only used fused quartz in one configuration, which itself developed only low interface stress values as compared to the use of a silicon substrate. The Petition contends “it was a well-known property that glass elements such as fused silica (*i.e.*, quartz) propagate and modify a stress wave to cause a

rarefaction shock in experimental laser techniques” citing to Ex. 1021. This conflating of fused silica and fused quartz fails because Gupta-1992 does not describe or even fairly suggest “shock development” in its use of fused quartz, let alone its ability to generate high tensile stresses at the test interface and no other document relied on by Petitioner discusses fused quartz. Likewise, the article “Shock-Wave Studies of PMMA, Fused Silica and Sapphire” (Ex. 1021) does not address fused quartz or its properties at all. Rather, Ex.1021 only describes the properties of “fused silica” and is silent on “fused quartz”. Again, confirming this, Petitioner’s expert’s own thesis, the Wang-Dissertation clearly states “[a]lthough shock development in fused silica was demonstrated in the 1970s by Barker and Hollenbach using a gas-gun facility, the usefulness of such shocks in applications to laser spallation has not been reported.” Ex. 1005, p. 81. Accordingly, at the time of writing the Wang-Dissertation and after careful consideration of Gupta-1992, Junlan Wang did not consider the “fused quartz” used with laser spallation experiments described in Gupta-1992 and “fused silica” used with laser spallation to be commensurate in properties and effect.

Gupta-1992 provides no evidence that fused quartz modifies a stress wave and the Petition provides no support based on the experimental results shown in Gupta-1992 that one of skill in the art would expect “fused quartz” to modify a stress wave.

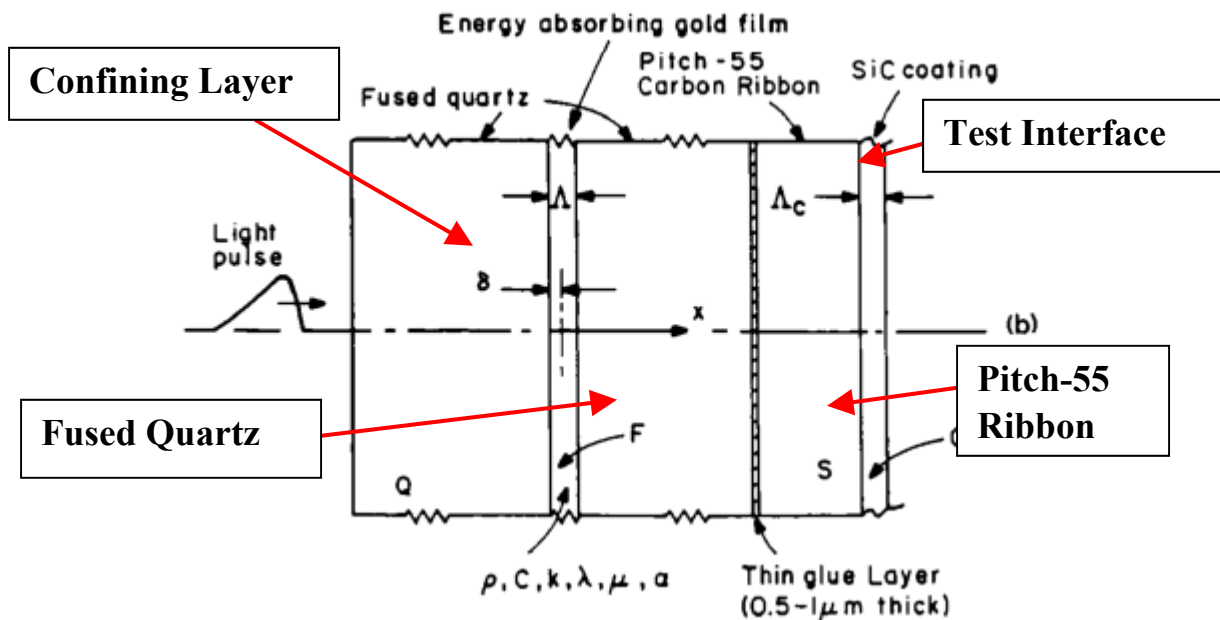
Under the first configuration shown below, both an Si crystal substrate (Si)/SiC coating interface and a Pyrolytic Graphite (PG) substrate/SiC coating interface were tested to determine the coating interface strength.



Annotated Fig. 2(a) from Gupta-1992

Experimental results indicated average interface strength for the Si/SiC system at the Si/SiC coating interface ranged from 3.7 to 10.88 GPa depending methods used to deposit the SiC coating. Ex. 1011, p. 175. For the PG/SiC system, the PG/SiC interface strength ranged from 3.68 to 7.48 GPa. No fractures in the Si or PG substrates were realized. Ex. 1011, p. 175.

In the second configuration shown below using the fused quartz/substrate/coating interface structure, the Pitch-55 ribbon/SiC interface strength was determined.



Annotated Fig. 2(b) from Gupta-1992

Experimental results indicated average interface strength for the Pitch-55/SiC system at the Pitch-55/SiC coating interface ranged from only 0.22 to 0.26 GPA. Ex. 1011, p. 175.

Nothing in these results indicates that the “fused quartz” modifies the stress wave or results in a rarefaction shock as detailed in the article “Shock-Wave Studies of PMMA, Fused Silica and Sapphire” (Ex. 1021), which is relied on by Boeing’s petition and Dr. Wang’s declaration to support their position. In fact, the configuration using fused quartz substrate bonded to the Pitch 55 Ribbon did not result in interface strength near the level of the Si crystal substrate or the Pyrolytic Graphite (PG) substrate systems due to failures in the Pitch-55 ribbon substrate. Consequently, all evidence from Gupta-1992 fails to support Dr. Wang’s contention that “it is anticipated based on the known knowledge of shock development in this material (Ex-1021) and this specific configuration” that the fused quartz substrate used in Gupta-1992 “is configured to propagate and modify a stress wave to the coating/substrate interface to generate a tensile stress between the coating” as alleged. Ex. 1003, p. 79; Pet. 49-50.

**2. The Petition Fails to Provide Any Support for the
Contention that Fused Quartz and Fused Silica Have the**

Same Properties and the Wang-Dissertation Supports the Opposite Conclusion

The Petition relies on three pieces of evidence to support its contention that Gupta-1992 teaches a fused quartz structure teaching the “glass element . . . configured to propagate and modify a stress wave to the coating/substrate interface” of independent claim 1 and the method step of “propagating and modifying a stress wave through the glass element to the coating/substrate interface”: (1) a mere conflating of “fused silica” and “fused quartz” throughout the Petition and Wang Declaration; (2) unsupported statements by Dr. Wang in the Wang Declaration; and (3) citation to Barker, L.M., Hollenbach, R.E. “Shock Wave Studies of PMMA, Fused Silica, and Sapphire,” J. Appl. Phys., 41, pp. 4208-4226) (Ex. 1021). As outlined below, it is clear that these basis fail to support Boeing’s contentions.

The naked conflating of the terms “fused quartz” and “fused silica as “fused quartz, *i.e.*, fused silica” is relied on in the Petition and the Wang Declaration as if these materials would somehow inherently exhibit the same properties. Pet., p. 50 and Ex. 1003, p. 81. Oddly, while conceding Gupta-1992 fails to describe the intended function or scientific mechanisms of fused quartz, Dr. Wang cites to Ex. 1021 for support. Yet, this journal article by Barker and Hollenbach only describes the function and scientific mechanisms of “high-purity fused silica”. This citation to an article discussing only the properties of high-purity fused silica to support the

contention that fused quartz would somehow have the same properties as fused silica lacks even a remote scintilla of logic.

Moreover, Dr. Wang's own statements in the Wang-Dissertation run counter to these *post hoc* rationalizations. The Wang-Dissertation cites to both Gupta-1992 and the Barker and Hollenbach article multiple times, yet relies on Gupta-1992 for its disclosure of laser-spallation methods, and the Barker and Hollenbach article for identifying rarefaction shocks in fused silica. *See, e.g.*, Ex. 1005, pp. 26, 40. In the context of this knowledge at the time of the Wang-Dissertation, Dr. Wang explained "[a]lthough shock development in fused silica was demonstrated in the 1970s by Barker and Hallenbach using a gas-gun facility, the usefulness of such shocks in applications to laser spallation has not been reported." Ex. 1005, p. 81. This objective evidence belies the Boeing and Dr. Wang *post hoc* rationalizations that one of skill in the art, at the time of the Wang-Dissertation, would have anticipated that fused quartz would or could have the same properties as a different material – fused silica.

C. Ground 3 – Wang-Dissertation in View of Gupta-1992

As is evident from the discussions of the Wang-Dissertation and Gupta-1992 above, Patent Owner submits that Boeing has failed to establish a likelihood of success in this ground asserting obviousness based on a modification of "fused silica" substrate embodiment in view of Gupta-1992's "fused quartz" / "Pitch-55

Ribbon” substrate embodiment. The Wang-Dissertation fails to enable the use of “fused silica” with an additional substrate, and Gupta-1992’s fused quartz/Pitch-55 ribbon exhibited substrate failure and did not produce adequate interface stresses due to this substrate failure, and thus fails to render obvious the following features of claims 1 and 13:

Claim 1

“a substrate having a thickness, said thickness defined by a first side and a second side in a first axis;”

“a glass element disposed on the second side of the substrate and axially spaced along the first axis;

wherein the glass element is configured to propagate and modify a stress wave to the coating/substrate interface to generate a tensile stress between the substrate and the coating.”

Claim 13

“a substrate, the substrate having a first side and a second side transversely disposed in a first axis”

“positioning a glass element along the first axis on the second side of the substrate;”

“propagating and modifying a stress wave through the glass element to the coating/substrate interface to generate a tensile force between the substrate and the coating;”

1. The Rationales Provided in the Petition

The Petition provide multiple rationales for modifying the Wang-Dissertation based on Gupta-1992:

Rationale (1) “[A] POSITA would have understood that Wang discloses that a variety of substrate/coating interfaces could be studied with the fused silica. Ex. 1003, ¶¶168-69. A POSITA would have envisaged a sample where a substrate/coating, such as silicon/aluminum, is positioned with the fused silica glass element on the second side to allow the adhesion strength of the interface to be studied, and Wang discloses this claim limitation as shown supra.11 *Id.*” Pet. , pp. 39-40.

Rationale (2) “To the extent PO argues that Wang does not disclose or suggest fused silica (“glass element”) on the second side of a substrate, it would have been obvious to a POSITA at the time of the invention to implement Wang’s fused silica glass element and sample configuration with a desired substrate as discussed in Gupta-1992. Ex. 1003, ¶¶170-74. Gupta-1992 is directed to tensile laser spallation techniques for thin films (Ex-1011, Abstract), so a POSITA would have had reason

to look to Gupta- 1992's teachings when implementing tensile laser spallation techniques and using fused silica to increase interfacial stress for testing thin films as taught by Wang. Ex. 1003, ¶171." Pet. p. 40.

Rationale (3) "A POSITA would have looked to Gupta-1992 for guidance as Gupta-1992 discloses using laser spallation techniques to test various substrate/film interfaces and discloses experiments for testing various substrates. Ex. 1011, Abstract, 142 ("[T]he technique can be used to determine the strength of planar interfaces between coatings ($>0.3\text{ }\mu\text{m}$ in thickness) and substrates in general"). Gupta-1992 shows bonding fused quartz to the second side of a desired substrate for testing. Id., Fig. 2(b). Wang discloses the same configuration for using fused silica to increase the interfacial failure in thin films and testing a variety of substrate/interfaces, by adding an extra layer of substrate. Ex. 1005, 81, 124; Ex. 1003, ¶172." Pet, pp. 40-41

Rationale (4) "A POSITA would have been motivated to combine Wang's advantageous use of fused silica and disclosure of adding an extra layer of substrate to test a variety of substrate/coating interfaces (Ex-1005, 124) with Gupta-1992's disclosure of testing different substrates, to position fused silica on the second side of a desired substrate/coating interface, and with a reasonable expectation of success, as Figure 2(b) shows how to create a sample with fused silica on the second side of a desired substrate. Ex. 1011, Fig. 2(b); Ex. 1003, ¶¶173-74. This would have been

a mere combination of known components and technologies, according to known methods, to produce predictable results. *Id.*; *KSR*, 550 U.S. at 416.” Pet, p. 41.

As is shown below, nowhere is there any support in the cited art that a POSITA would have the requisite expectation of success in using “fused silica” with Gupta-1992’s Pitch-55 Ribbon.

**2. The Wang-Dissertation Does Not Enable Using Fused Silica
in Combination with a Substrate to Test Interfacial
Strength**

As discussed *supra* section VII. A. 4. a.-f, the Wang-Dissertation fails to enable a POSITA to make and use a fused silica with an additional substrate disposed between the fused silica and the test coating. The clear evidence in the Wang-Dissertation shows clear problems caused when using a silicon substrate – damage to the substrate and stress attenuation. While the Wang-Dissertation enables a POSITA to use a fused silica substrate, or alternatively, a silicon substrate, directly bonded to a test film of Al, it is clear from the testing results of silicon and fused silica that a POSITA would not expect that a combination of fused silica – an additional substrate – test film combination would successfully produce the critical interface stresses needed to load the test film interface. The general arrangement tested in the Wang-Dissertation is shown below:

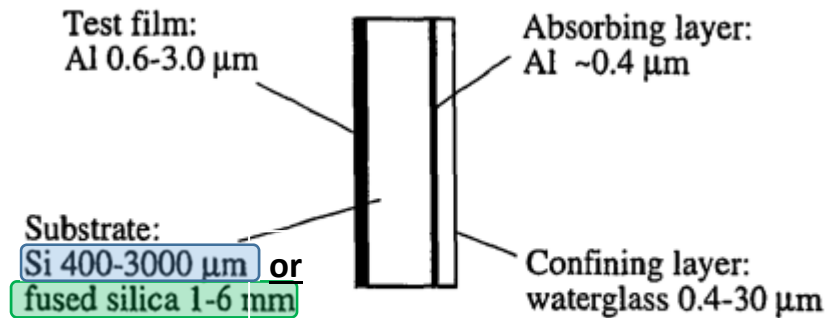


Figure 3.2: Schematic of thin film sample for spallation experiment.

Ex. 1005, pp. 44-45 (annotated).

The Wang-Dissertation emphasizes the “particular importance” of fused silica substrates due to their negative non-linear elasticity that causes the compressive stress was to “evolve a decompression shock, which is **critical for generation of fast fall times needed for significant loading of the thin-film interfaces.**” Ex. 1005, p. iv. (emphasis added). The dissertation also describes that “stress at the film interface can be much less than the stress acting on the substrate because the nearby free surface releases some of the stress.” Ex. 1005, p. 21. In view of the well documented problems with stress attenuation and substrate damage within the Wang-Dissertation itself, there is no logical basis to support the conclusion that a POSITA would expect the combination of fused silica, which creates a decompression shock, with another substrate and test film would function to provide these critical stresses to load the test interface without substrate damage. Neither Boeing nor their expert Dr. Wang addressed any of these documented issues. To the

contrary, their basis for obviousness are built on a mere stacking of components together without any technical reasoning in view of the problems and difficulties created by substrate damage and substrate stress attenuation.

a. The Wang-Dissertation Only Described Substantial Loading at the Test Interface without Damage When Using a Single Fused Silica Substrate

As noted in the dissertation, the **fused silica substrate was not damaged** by the tensile stress. Ex. 1005, p. 51, 81 and 120. The Wang-Dissertation attributed this result to fused silica being intrinsically strong such that no substrate damage occurs before interfacial failure. *Id.* at p. 81. However, at increased substrate thickness, even the fused silica exhibited substantial stress attenuation as shown in Table 3.4 reproduced below (Ex. 1005, p. 69):

Table 3.4: Effect of substrate thickness on $\sigma_{\text{sub}}^{\text{max}}$ and $\sigma_{\text{int}}^{\text{max}}$ on fused silica samples with a 1.1 μm thick aluminum film at maximum YAG power (0.140 J/mm²).

Substrate thickness	1500 μm	3000 μm	6000 μm
$\sigma_{\text{sub}}^{\text{max}}$	-1.2	-0.5	-0.3
$\sigma_{\text{int}}^{\text{max}}$	370	350	50

While the loading is substantial at the interface for fused silica thicknesses of 1500 μm and 3000 μm at 370 MPa and 350 MPa, respectively, the interface stress dropped dramatically at a thickness of 6000 μm to only 50 MPa. The logical

conclusion here is clear, substrate thickness substantially impacts interface stress and highly impacts the “critical” effects of the “decompression shock” needed for loading of the thin-film interfaces. Ex. 1005, p. iv. The Wang-Dissertation describes that “significant attenuation in the peak stress occurs as the stress travels through longer distances.” Ex. 1005, p. 38. Even more compelling is that the Wang-Dissertation describes that this “attenuation associated with the shock wave is even greater than that associated with geometric attenuation.” *Id.* Absent is any discussion of how wave attenuation can be minimized when adding an additional substrate between the fused silica and the test film. Notably absent in the Wang-Dissertation, the Boeing Petition, or the Wang Declaration is there any logical reasoning to overcome this clear irrefutable evidence or to establish any expectation of success in coupling another substrate to the fused silica.

Even further, the Wang-Dissertation provides substantial evidence showing damage is typical in the silicon substrate. Ex. 1005, pp. 47 and 62. Silicon substrates at a thickness of 600 μm were tested and substrate stress values were plotted in FIG. 3.14(c):

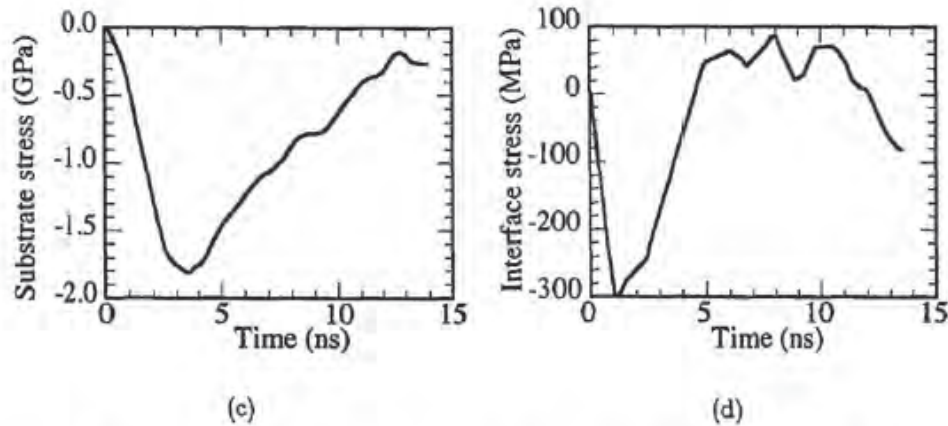


Figure 3.14: A typical data processing cycle for a 1.0 μm aluminum film deposited on a 600 μm thick silicon substrate (laser fluence of 0.140 J/mm²): (a) interferometric fringe pattern; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile.

The maximum interface tensile stress reached 60 MPa. Yet, as noted by the Wang-Dissertation, silicon substrate damage is typical. Ex. 1005, pp. 47 and 62. As concluded in the Wang-Dissertation “[o]bservations of damage revealed both substrate and film failure in silicon substrate samples, but only film failure in fused-silica substrates samples.” Ex. 1005, p. 81. Thus, fused silica was identified as “an ideal material for studying the mechanics of the spallation test in that it is intrinsically strong and no substrate damage occurs before interfacial failure.” *Id.* Absent from the Wang-Dissertation is any teaching or disclosure of how use of a fused silica, which creates a decompression shock, with an additional substrate without experiencing damage. Notably, the Wang-Dissertation only attributes this lack of damage in the fused silica to its intrinsic strength.

Accordingly, the Wang-Dissertation fails to enable the forward looking “Future Work” statement that “[b]y inserting an extra layer of the desired substrate material between the fused silica and the thin film, the current technique can be used to test a variety of substrates and films.”

**b. The Petition’s Reliance on Gupta-1992 Ignores the Failed
Results of the Relied on Configuration**

The Petition relies on a structure from Gupta-1992 where fused quartz is bonded to a second side of the substrate for testing. Pet., p. 40-41. As a basis for modifying the Wang-Dissertation based on Gupta-1992, the Petition contends that a POSITA would be motivated “to position fused silica on the second side of a desired substrate/coating interface, and with a reasonable expectation of success, as Figure 2(b) shows.” Pet., p. 41.

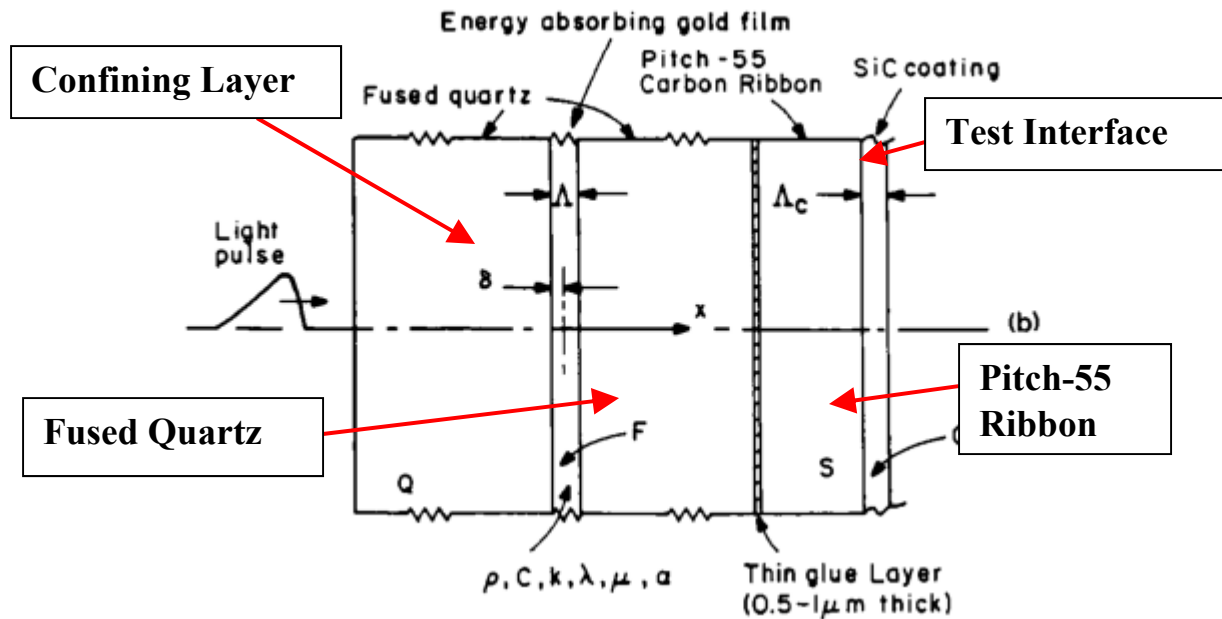
Noting that “[i]nterfacial adhesion is a critical parameter governing the mechanical behavior and reliability of a thin film on a substrate”, the Wang-Dissertation studied laser spallation methods to load the interface in a non-contacting manner to study thin-film adhesion. Ex. 1005, p. 3. In contrast to this purpose and goal, the Petition seeks to establish obviousness by modifying the embodiment where fused silica is directly bonded to the test film by relying on the structure of Fig. 2(b) of Gupta-1992. Pet., p. 70. The basis for this modification is that Gupta-1992 discloses testing different substrates by positioning fused silica on the second

side of a desired substrate/coating interface with a reasonable expectation of success.

Pet., p. 41; Ex. 1003, p. 70.

These contentions belie the express disclosures of the Wang-Thesis and Gupta-1992. As noted above, the Wang-Dissertation only enables the use of fused silica when directly attached to the test film. Additionally, the Wang-Dissertation outlines issues with a silicon substrate, i.e., damage and stress attenuation, which do not permit those systems to develop adequate loading at the test interface either due to failure of the substrate or due to attenuation within the substrate. It is also worthwhile to note that Gupta-1992 does not use fused silica, but fused quartz. As noted previously, neither the Petition nor the Wang Declaration establish these two different materials exhibit the same material properties.

Gupta-1992, on the other hand, does show the following configuration that includes fused quartz bonded to Pitch-55 Ribbon:



Annotated Fig. 2(b) from Gupta-1992

However, in this configuration, the Pitch-55 substrate experienced damage and did not generate the interface loading experienced in the other single substrate models tested using Si Crystal (Si) or Pyrolytic Graphite (PG) Substrates, which did not use the fused quartz. Experimental results indicated average interface strength for the Si/SiC system at the Si/SiC coating interface ranged from 3.7 to 10.88 GPa depending methods used to deposit the SiC coating. Ex. 1011, p. 175. For the PG/SiC system, the PG/SiC interface strength ranged from 3.68 to 7.48 GPa. No fractures in the Si or PG substrates were realized. Ex. 1011, p. 175. Experimental results indicated average interface strength for the fused quartz/Pitch-55/SiC system at the Pitch-55/SiC coating interface ranged from only 0.22 to 0.26 GPa. Ex. 1011, p. 175. Gupta-1992 explains that these results were governed by fractures within

the Pitch-55 ribbon itself. *Id.* As explained in the Wang-Dissertation, damage patterns are not desired in laser spallation experiments as they result in causing the test film to come off the substrate before the interface fails. Ex. 1005, pp. 47, 61. This ground is relying on a failed experiment to somehow turn that into some sort of expectation of success by completely ignoring substrate damage issues.

Consequently, Gupta-1992 does not provide any expectation of success. Rather, to the contrary, the embodiment relied on by Boeing failed to produce the requisite interface loading and experienced substrate damage. The evidence of record fails to support any reasonable expectation of success or predictable results. The contentions laid out in the Petition and the reasoning in the Wang Declaration is void of any logical articulation as to why these problems of attenuation and substrate damage would not occur. Instead, the support is provided in the way of naked contentions of predictability and expectation of success, with no consideration of these problems or how a POSITA would resolve them.

D. Ground 7 – Wang-2002 in view of Gupta-1992

This ground parallels the Wang-Dissertation and Gupta-1992 ground (Ground 3), with the exception that Wang-2002 does not provide the non-enabled “Future Work” statement relied on in Ground 3. Wang-2002 describes the same test configuration and as is described in the Wang-Dissertation and alternatively tests fused silica and silicon at the base substrate that is bonded directly to the test film

(A1). *Compare* Ex. 1003, Figs. 2.1, 3.1 and 3.2 *with* Ex. 1016, Figs. 1, 2 and 3. The experimental results are also the same as reported in the Wang-Dissertation. *Compare* Ex. 1005, Figs. 3.14, 3.15 and 3.16; and Tables 3.2, 3.3 and 3.4 *with* Ex. 1016, Figs. 6, 8 and 9; and Tab 2, 3 and 4.

Otherwise, Wang-2002 identifies the similar problems identified in the Wang-Dissertation (Ex. 1005): (1) attenuation of stress due to the thickness of the substrate; (2) realized tensile stresses at the test interface being lower than the stress acting on the substrate as the free surface of the test film releases some of the stress; and (3) damage to the substrate. Ex. 1016, p. 76-77.

As set forth below, the Petition fails to provide a likelihood of success of establishing obviousness of the following features of claims 1 and 13:

Claim 1

“a substrate having a thickness, said thickness defined by a first side and a second side in a first axis;”

“a glass element disposed on the second side of the substrate and axially spaced along the first axis;

wherein the glass element is configured to propagate and modify a stress wave to the coating/substrate interface to generate a tensile stress between the substrate and the coating.”

Claim 13

“a substrate, the substrate having a first side and a second side transversely disposed in a first axis”

“positioning a glass element along the first axis on the second side of the substrate;”

“propagating and modifying a stress wave through the glass element to the coating/substrate interface to generate a tensile force between the substrate and the coating;”

Neither Wang-2002 nor Gupta-1992 disclose or suggest using a “glass element” that modifies a stress wave in combination while be disposed on the second side of a substrate – where the coating is applied to the first side of the substrate. Wang-2002 only studied two substrates directly bonded to the testing film, fused silica and silicon. Never were these used in combination. Gupta-1992 tested a configuration where a fused quartz/Pitch-55 carbon ribbon substrate/ SiC coating structure was tested. However, there is no disclosure that fused quartz modifies the stress wave and this test was unsuccessful as the Pitch-55 carbon ribbon structure fractured during testing.

1. The Rationales Provided in the Petition

The Petition provide multiple rationales for modifying the Wang-2002 based on Gupta-1992:

Rationale (1) – “Based on these teachings, a POSITA would have understood that Wang-2002 discloses that a variety of substrate/coating interfaces could be studied with the fused silica. *Id.*, Fig. 3; Ex. 1003, ¶244. A POSITA would have envisaged a sample where the “substrate” (such as a silicon/coating interface) is positioned with the fused silica “glass element” on the second side to allow the adhesion strength of the interface to be studied, and Wang-2002 discloses this claim limitation. *Id.*” Pet., p. 64; *see also* Ex. 1003, p. 98.

Rationale (2) – “A POSITA would have been motivated to combine Wang-2002’s disclosure of the advantages of fused silica with Gupta-1992’s disclosure of testing different substrates to position the fused silica on the second side of a desired substrate/coating interface, and with a reasonable expectation of success, as Gupta-1992 in Figure 2(b) shows how to create a sample with fused silica on the second side of a desired substrate. *Id.*; Ex. 1011, Fig. 2(b); Ex. 1016, Fig. 3. This would have been a mere combination of known components and technologies, according to known methods, to produce predictable results” Pet., p. 65-66; *see also* Ex. 1003, p. 100.

2. Considering the Art as a Whole Neither Wang-2002 Nor Gupta-1992 Provide Support for Petitioner’s Rationales

Wang-2002 only tests a fused silica bonded directly to a test film or a silicon substrate directly bonded to a test film. Notably, the Rationale (1) provided by

Petitioner seeks to add fused silica to the second side of the silicon substrate on the basis that Wang-2002 identifies fused silica “is an ideal material for studying mechanics of the spallation test in that it is intrinsically strong and the shock mechanism is advantageous for testing thin-film interface adhesion. Ex. 1003, p. 98. The Wang Declaration further concludes that “a POSITA would have understood that I disclosed that a variety of substrate/coating interfaces could be studied with the fused silica.” Ex. 1003, p. 98. These contentions only find support from “fused silica” testing as the substrate bonded to a test film as shown in Fig. 3 reproduced below.

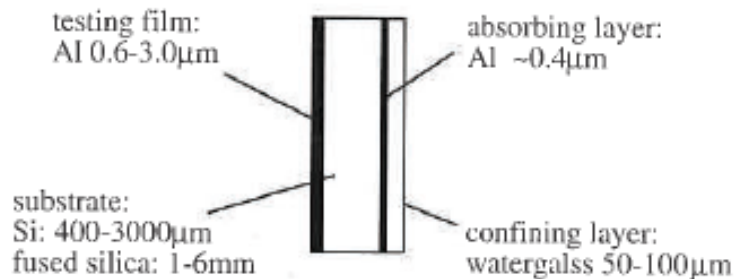


Fig. 3—Schematic of thin film sample for spallation experiment

Fig. 3 of Wang-2002, p. 76

Nowhere was “fused silica” tested in combination with an additional substrate. Notably, the success of the fused silica is attributed to it being “intrinsically strong”. Ex. 1003, p. 98. What is not shown by the Petition or the

Wang Declaration is how a different substrate, such as a silicon substrate (in the case of Wang-2002) or a Pitch-55 Ribbon substrate (in the case of Gupta-1992) could withstand the shock formation induced by the fused silica or the attenuation problem impacting interface stress at the substrate/test film interface. The Wang-2002 article outlines the problems of stress attenuation and substrate damage, but fails to consider or explain how fused silica overcomes any of these problems, let alone how the shock formation would not induce more damage in the substrate or suffer from substrate attenuation. Instead, Dr. Wang merely provides that a “POSITA would have envisaged a sample where the ‘substrate’ (such as a silicon/coating interface) is positioned with the fused silica “glass element” on the second side to allow the adhesion interface to be studied.” Ex. 1003, p. 98. Clearly lacking from this conclusory statement is how a POSITA would find the requisite expectation of success in making this untested combination, especially in view of the known problems of substrate damage and stress attenuation through substrates. Neither of these problems are addressed.

a. Significant Problems in Tested Silicon Substrates as Compared to Fused Silica Substrates

In the experiments covered in Wang-2002, only fused silica showed successful results, i.e., high interface stress and no substrate damage. In the case of fused silica as the substrate, the maximum interface **stresses realized at the**

coating/substrate interface were high as desired – 350 to 370 MPa at thicknesses of 3000 μm and below. Ex. 1016, p. 81. The **fused silica substrate was not damaged** by the tensile stress. Ex. 1016, pp. 77 and 82. Because the fused silica is intrinsically strong, no substrate damage occurs before interfacial failure. *Id.* at p. 82.

In contrast, when using silicon as the substrate, tests **revealed substrate damage and high stress wave attenuation** - only realizing a stress of 75 MPa at a thickness of 3000 μm . Ex. 1016, p. 81. Additionally, Wang-2002 observed that maximum substrate stress and maximum interface stress become lower for greater substrate thicknesses. Ex. 1016, p. 81; Tables 3 and 4.

**b. Silicon Substrates Exhibit Damage at Relatively Low Tensile
Stress Without Fused Silica**

In the case of silicon substrates, quantitative measurements of displacement, substrate stress and interface stress were performed. Ex. 1016, p. 77-78. Silicon substrates at a thickness of 600 μm were tested and substrate stress values were plotted in Fig. 6-A, (c) and (d):

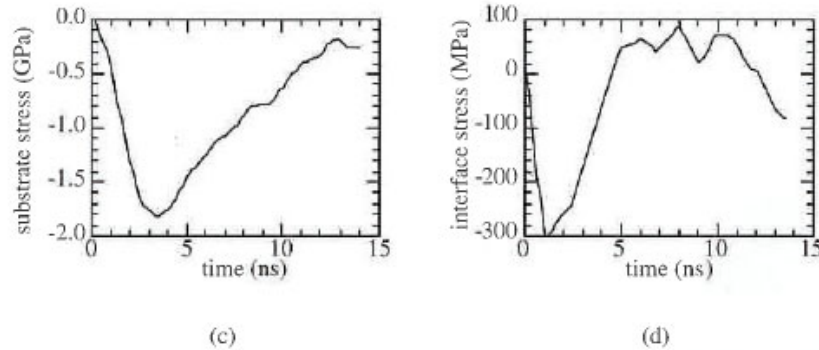


Fig. 6—A typical data processing cycle for a 600 μm thick Si substrate sample: (a) interferometric fringe pattern; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile

The maximum interface tensile stress reached 80 MPa. Yet, as noted by the Wang-2002, silicon substrate damage occurs due to cleavage under action of the tensile wave. Ex. 1016, p. 77.

**c. Fused Silica produces Much Higher Tensile Stresses Further
Inducing Damage in a Coupled Substrate**

Similar tests showed quantitative measurements of displacement, substrate stress and interface stress in both the fused silica and silicon substrates. Ex. 1016, pp. 78-80. Effects of substrate thickness were also tested. *Id.* at pp. 80-82. As shown in Tables 3 and 4, for a 3000 μm thick substrate, the maximum substrate stresses were similar at -0.55 for silicon and -0.5 GPa for fused silica, but maximum interface stresses were much lower in the silicon substrate at 75 MPa as compared to 350 MPa for the fused silica substrate.

TABLE 3—EFFECT OF SUBSTRATE THICKNESS ON Si SUBSTRATE SAMPLES

Substrate thickness (μm)	400	600	3000
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	-1.7	-1.4	-0.55
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	100	90	75

TABLE 4—EFFECT OF SUBSTRATE THICKNESS ON FUSED SILICA SAMPLES

Substrate thickness (μm)	1531	3063	6125
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	-1.2	-0.5	-0.3
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	370	350	50

These fused silica substrates were tested at 3000 μm thickness and the results were plotted in Fig. 8. These tests showed significant differences due to the nonlinear properties of the fused silica and resulted in higher tensile stresses. Notably, the laser fluence used in both the silicon substrate and the fused silica substrate tests were the same at 0.14 J/mm². However, the interface stress realized when using the fused silica substrate was an order of magnitude higher at about 0.6 GPa (600 MPa) as shown in Fig. 8 reproduced below:

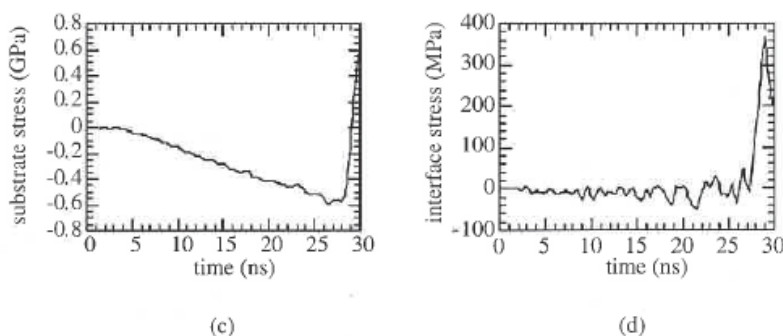


Fig. 8—Typical data profile for a fused silica (3063 μm thick) substrate sample: (a) interferometric fringe pattern, arrow points to turning point; (b) displacement profile; (c) substrate stress profile; (d) interface stress profile

As the Wang-2002 reports that damage occurs in silicon substrates, it becomes apparent that using a fused silica substrate in combination with a substrate would also lead to higher tensile stresses and would lead one of skill to expect more substrate damage in such a coupled system. Wang-2002 teaches that because the purpose is to measure adhesion between the film and substrate, substrate cleavage is not desired. Ex. 1016, p. 77.

d. Substrate Attenuation Significantly Impacts Test Interface Stress

The Wang-2002 provides clear results using only a fused silica substrate bonded directly to a test film. No insights or evidence are provided to enable one of skill in the art to modify this structure to add an additional substrate.

As experiments show, the significant nonlinear properties of the fused silica will “cause the compressive stress wave generated by the pulsing laser to evolve a decompression shock.” However, as shown in Table 3, substrate thickness substantially impacts the maximum interface stress, even in fused silica:

TABLE 3—EFFECT OF SUBSTRATE THICKNESS ON SI SUBSTRATE SAMPLES

Substrate thickness (μm)	400	600	3000
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	-1.7	-1.4	-0.55
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	100	90	75

TABLE 4—EFFECT OF SUBSTRATE THICKNESS ON FUSED SILICA SAMPLES

Substrate thickness (μm)	1531	3063	6125
$\sigma_{\text{substrate}}^{\text{max}}$ (GPa)	-1.2	-0.5	-0.3
$\sigma_{\text{interface}}^{\text{max}}$ (MPa)	370	350	50

As these results show, greater silicon substrate thickness shows increased stress attenuation. At the same tested conditions for a 3000 μm thick substrate, while maximum substrate stresses were comparable (-0.55 MPa versus -0.5 MPa), maximum interface stresses were much lower after the compression wave is passed through the silicon substrate.

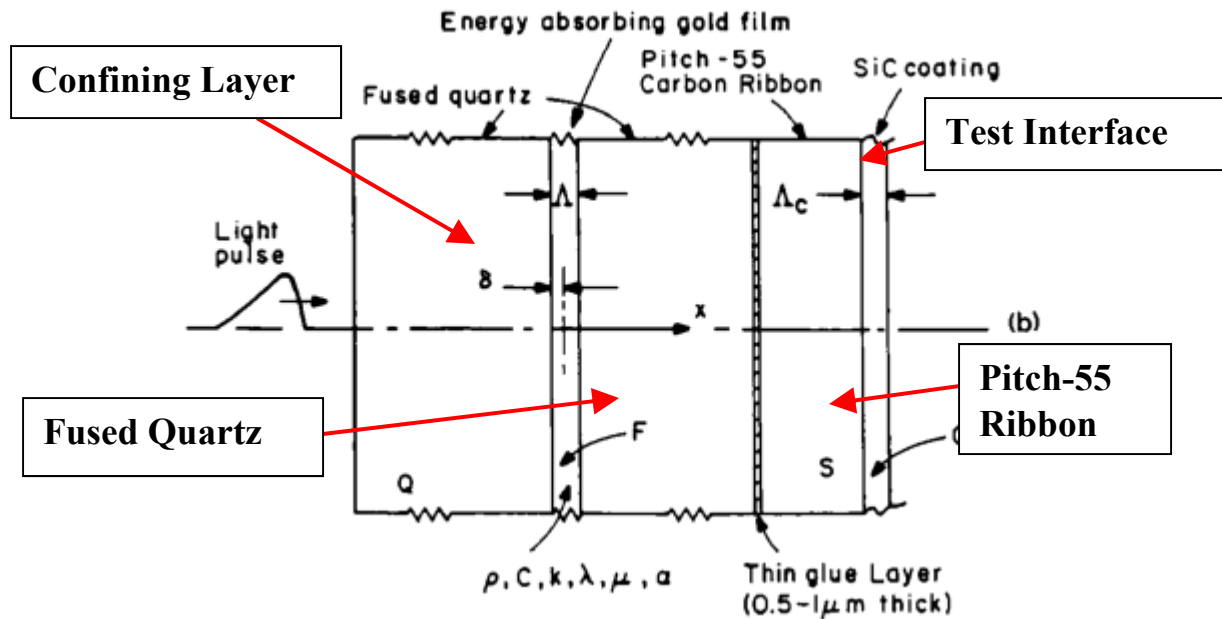
While the Wang-Dissertation enables one of skill in the art to generate adequate tensile stresses at the substrate/test film interface of 350-370 MPa when using fused silica, the clear evidence of stress wave attenuation show significant decreases in tensile stress when the same laser energy is applied to a silicon substrate. This impact is nearly a 5X drop at 75MPa after the compression wave travels through the silicon substrate. In view of this evidence, absent is any teaching or disclosure in the Wang-Dissertation to enable one of skill in the art to reach the necessary tensile stress of 350-370 MPa reached in fused silica by further transferring the same

energy through a subsequent substrate as outlined in the “Future Work” section of the Wang-Dissertation.

It is also worthwhile to note that the Wang-Dissertation describes that “significant attenuation in the peak stress occurs as the stress travels through longer distances.” Ex. 1005, p. 38. Even more compelling is that the Wang-Dissertation describes that this “attenuation associated with the shock wave is even greater than that associated with geometric attenuation.” *Id.* Absent is any discussion of how wave attenuation can be minimized when adding an additional substrate between the fused silica and the test film. Accordingly, there is not support that a POSITA would have the requisite expectation of success in making this combination.

e. Gupta-1992 - Substrate Damage

In the relied on structural configuration, interface strengths were tested for the Pitch-55 ribbon/SiC interface using the structure of Fig 2(b) as shown below:



Annotated Fig. 2(b) from Gupta-1992

Having a different structure than used in Fig. 2(a), testing of the Pitch-55/SiC coating interface additionally included a fused quartz substrate between the confining layer and the Pitch-55 Carbon Ribbon as shown in Fig. 2(b). Experimental results indicated average interface strength for the fused quartz/Pitch-55/SiC system at the Pitch-55/SiC coating interface ranged from only 0.22 to 0.26 GPA. Ex. 1011, p. 175. Gupta-1992 explains that these results were most likely governed by fractures within the Pitch-55 ribbon itself. *Id.* The Petition and Wang Declaration provide conclusory rationales, but ignore the issues of substrate damage. This substrate damage is described in both references. While it is convenient for the challenger to ignore the “damage” apparent in both Wang-2002’s silicon substrates as well as Gupta-1992’s Pitch-55 Ribbon, these are important considerations and

problems that show Boeing's naked conclusory basis for asserting obviousness are unsupported and not enough to establish the requisite likelihood of success of this ground.

CONCLUSION

For at least the foregoing reasons, Boeing's Petition fails to establish the required reasonable likelihood of success with regard to Grounds 1-7. Therefore, the Petition should be denied in its entirety, and no trial instituted.

Respectfully submitted,

/Chid S. Iyer/

Chidambaram S. Iyer, Reg. No.: 43,355

Raja N. Saliba Reg. No. 43,078

David P. Emery, Reg. No. 55,154

Mark Boland, Reg. No. 32,197

John T. Callahan, Reg. No. 32,607

SUGHRUE MION, PLLC

2000 Pennsylvania Ave NW, Suite 9000

Washington, DC 20006

Telephone: 202-293-7060

Facsimile: 202-293-7860

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/Chid S. Iyer/

Chidambaram S. Iyer, Reg. No.: 43,355

Raja N. Saliba Reg. No. 43,078

David P. Emery, Reg. No. 55,154

Mark Boland, Reg. No. 32,197

John T. Callahan, Reg. No. 32,607

SUGHRUE MION, PLLC

2000 Pennsylvania Ave NW, Suite 9000

Washington, DC 20006

Telephone: 202-293-7060

Facsimile: 202-293-7860

Date: JULY 10, 2024

CERTIFICATE OF SERVICE

The undersigned certifies that a copy of the attached Patent Owner Preliminary Response is being served by electronic mail on the following counsel of record:

/Chid S. Iyer/

Chidambaram S. Iyer, Reg. No.: 43,355

Raja N. Saliba Reg. No. 43,078

David P. Emery, Reg. No. 55,154

Mark Boland, Reg. No. 32,197

John T. Callahan, Reg. No. 32,607

SUGHRUE MION, PLLC

2000 Pennsylvania Ave NW, Suite 9000

Washington, DC 20006

Telephone: 202-293-7060

Facsimile: 202-293-7860

Date: July 10, 2024