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EFFECT OF DISCONTINUITIES AS A MEANS TO ALLEVIATE THERMAL-EXPANSION MISMATCH DAMAGE IN LAMINAR COMPOSITES

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16. Abstract

An investigation of Nichrome/Tungsten laminar composites showed that intentionally introduced discontinuities, such as perforations through or grooves on the surface of the matrix laminae, improved thermal expansion mismatch damage resistance. It was found that specimens having smooth matrix laminae surfaces were virtually destroyed by delamination in 21 or fewer fast cool cycles in which they were water quenched from 981° C. Specimens having interior matrix laminae with discontinuities and relatively thin, nondiscontinuous, surface matrix laminae resisted 50 similar cycles without evident delamination damage.

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SUMMARY

An investigation was conducted to determine if discontinuities such as perforations through or a grid pattern of grooves on the breadth surfaces of the matrix laminae in laminar composites would improve their thermal expansion mismatch damage resistance. Laminar composite specimens having tungsten reinforcement sheets and Nichrome matrix sheets were thermally cycled. A number of thermal cyclic tests were used. One was a fast cool cycle that consisted of placing specimens into an 981° C preheated furnace, retaining them in the furnace for 1/2 hour and then water quenching them. Another was a slow-ccol cycle and consisted of placing specimens into a vacuum hot press furnace, heating them to 981° C in about two hours, retaining them at that temperature for 1/2 hour and then shutting off furnace power, allowing the specimens to cool gradually (4 hr) to room temperature. The third test consisted of placing specimens into a 981° C perheated furnace, retaining them in the furnace for 1/2 hour, then removing them to allow them to air cool. These specimens cooled to 60° C in about 20 minutes.

It was found that standard specimens having smooth matrix laminae surfaces and uniformly thick (0.051 cm) laminae began to delaminate after two or three fast cool cycles; the specimens were virtually destroyed by delamination in 21 cycles or fewer. Specimens having perforations in the matrix laminae or an impressed groove pattern on the matrix laminae had greater resistance. Although the outermost surface laminae became semidetached, the remaining laminae were undelaminated after 25 and 50 cycles, that is, upon conclusion of the tests for the respective specimen configuration.

When relatively thin (0.003 cm) surface laminae were used in addition to the grid indentation modified matrix laminae, these modified specimens withstood 50 fast cool thermal cycles with no sign of delamination damage at which point the tests were stopped.

INTRODUCTION

Composite materials consisting of reinforcing elements within a matrix are being studied for use in high strength/weight applications. Composite materials often contain constituents having disparate coefficients of thermal expansion. This difference in expansivity gives rise to "thermal expansion mismatch." Under such a condition the composite constituents are restrained from reaching the dimensions they would have attained had they been able to expand or contract freely during heating and cooling. This restraint gives rise to internal stresses and strains which can degrade the structural integrity of the composites, adversely affect the mechanical properties of the composites and/or produce thermal fatigue fracture (ref. 1). Additional investigations of the effect of thermal expansion mismatch are reported in references 2 to 8.

Thermal expansion mismatch effects can be particularly severe in laminar composites in that the occurrence of matrix-reinforcement unbonding can result in gross macro-structural failure of the composite, that is, delamination. It was felt that in the case of laminar continuous sheet or foil composites, to which this paper is limited, that the introduction of discontinuities on the surfaces of the matrix laminae would have beneficial effects insofar as overcoming the thermal mismatch problem is concerned. On the basis of a simple analysis, it was believed that small discrete matrix segments would produce short shear lengths which would lead to lessened internal shear stress and a reduced tendency towards delamination and material degradation. It was thought that continuous matrix elements having surface discontinuities could approximate this effect and be easier to assemble into composites and in addition it was also believed that discontinuities would promote interlaminar bonding. The latter effect could result because excess matrix material resulting from surface irregularities on adjacent laminae could readily flow into nearby matrix discontinuities.

The objective of this study was to determine if the intentional introduction of discontinuities on the bonded surfaces of the matrix laminae in laminar composites would improve thermal expansion mismatch damage resistance; composite delamination was taken as the measure of damage.

The materials used in this study were tungsten (reinforcement) and Nichrome (matrix) since they had respective coefficients of thermal expansion similar to those that would be found in a great variety of more practical systems. Rather thick, that is, 0.051 cm, laminae were generally used because it was found in a previous study in which tungsten and Nichrome were used (ref. 1), that this thickness resulted in thermal expansion mismatch damage. Specimens were either slow cooled or fast cooled from 981° C to ambient temperature using three different thermal cyclic tests. The specimens were examined for macro-structural degradation.

MATERIALS AND PROCEDURE

Materials

Commercially available tungsten and Nichrome were used; the former material was considered to be the reinforcement and the latter the matrix. The tungsten was all nominally 0.051 cm thick. The Nichrome laminae used in the interior of all the specimens were also nominally 0.051 cm thick; the surface Nichrome laminae were 0.051 cm thick in some specimens and were 0.003 cm thick in others. The laminae were all nominally 2.54 by 7.62 cm in the plane dimension.

Specimens

The Nichrome and tungsten were alternately layered and generally each composite had six matrix laminae and five reinforcement laminae.

Hot pressing was done at 981° C at a pressure of 13.8 MN/m^2 applied for 4 hours; the furnace power was then shut off and the specimens allowed to cool of their own accord. Details of specimen preparation are described further in reference 2. The specimens were used as they came from the hot press, that is, the edges were not ground smooth. This was done to expedite production of specimens and to avoid possible bond damage. There was a slight gap between adjacent laminae around the perimeters of the specimens. This was due to a slight compressive deformation around the perimeters of the Nichrome laminae after they had been sheared to size. Visual examination of specimens that had delaminated during testing indicated that there were unbonded zones about 0.07 cm wide around the perimeters of the specimens; the remaining interior portions of the laminae appeared to have been well bonded. Furthermore, all metallographic specimens that were viewed, indicated that the bonds were satisfactory.

Discontinuities were introduced into some of the matrices by drilling a pattern of 0.476 cm diameter holes into these laminae. A perforated matrix lamina is illustrated in figure 1(a). The hole size and pattern used was arbitrary. It was desired that a large number of discontinuities be introduced. Discontinuities were introduced into other matrix laminae by impressing a grid pattern of grooves on the breadth surfaces of these laminae. The grid was 0.635 cm square and the indentations were about 0.013 cm deep (fig. 1(b)). The grid pattern and depth of penetration were arbitrary and had the objective of obtaining a large number of effective discontinuities. The grid pattern was impressed using a set of dies.

A number of specimen types were used. Those having matrices with smooth surfaces were designated as standard; those having matrix surface discontinuities were designated as modified. These specimen types are listed in table I, and illustrated in figures 2(a) to 2(e).

Test Cycles

One fast cool and two types of slow cool thermal cycle tests were run. The former test would be more representative of what might occur in a practical application. The latter tests would tend to permit relaxation to occur and thus reduce stress after cool down; however, they could also introduce relatively more plastic strain after cool down, than would occur in a fast cool test. It has been reported in reference 9 that plastic strain occurring in low cycle fatigue can be damaging.

The fast cooled thermally cycled specimens were placed into commercially available thin walled (i.e., 0.0051 cm) heat treating envelopes which were sealed by folding. A small bit of alumina powder was placed into each envelope to prevent contact of specimens with the envelope. In general, one modified and one standard specimen were run concurrently in the same envelope. The envelopes became embrittled after each cycle and were replaced after each cycle. The specimens were inspected after each thermal cycle.

The fast cool thermal cycle specimens were inserted into a 981° C preheated muffle furnace, retained in the furnace for 1/2 hour and then quenched into a container of water. It was estimated that it took about 10 minutes for the specimens to reach temperature equilibrium at 981° C. When quenched, all evolution of steam ceased after about 2 minutes so that the specimens were cooled to 100° C or less in about 2 minutes. After each water quench, the specimens were dried under a heat lamp at about 200° C for 1/2 hour to completely evaporate any water.

One type of slow cool thermal cycle test was performed in the vacuum hot press (with rams retracted). In general, a modified and a standard specimen were tested concurrently. They were inspected after each cycle. This slow cool cyclic test consisted of heating the specimens to temperature in about 2 hours, maintaining that temperature for 1/2 hour, and shutting off furnace power. The specimens remained in the hot press overnight. It is estimated that the specimens cooled to essentially room temperature in about 4 hours. The specimens were removed and inspected after each thermal cycle.

A second type of slow cool thermal cycle test was performed in air on some specimens. However, with the exception of one standard I-A specimen, oxidation of the tungsten occurred, obscuring the results. The test consisted of heating specimens (in envelopes) in the 981° C preheated muffle furnace for 1/2 hour and removing them from the furnace and placing them on a refractory brick. It took 20 minutes to cool to 60° C. The specimens were inspected after each thermal cycle and then placed into a new envelope for each additional thermal cycle.

The criterion used for failure was not a rigorous one. In the instance of the fast cooled standard specimens, they were thermally cycled until they were virtually destroyed by delamination even though they had already begun to delaminate after two or three cycles. An objective was to determine whether delamination was progressive. In the instance of the fast cooled modified specimens, they were initially run for 25 cycles and later for 50 cycles, even though the surface laminae were partially delaminated after several cycles.

A similar rationale was used for continuing the slow cool tests even though delamination was initiated after only several thermal cycles.

The maximum number of cycles applied for either the fast cool or slow cool type of test or for any specimen design was 50.

RESULTS AND DISCUSSION

Fast Cool Cycles

Standard I-A Specimens. - From table II, it can be seen that all four of the specimens tested grossly delaminated after 21 cycles or less. Some delamination occurred in each specimen after the first two or three cycles. The delamination was progressive with additional damage occurring at successive cycles. A typical specimen at cessation of thermal cyclic testing is shown in figure 3.

Modification I-A specimens. - The modification I-A specimens represented the first attempt to improve the thermal cyclic delamination resistance of the laminar composite specimens. The matrices used had a pattern of perforations drilled in them as shown in figure 1(a).

As may be seen from table II, all five of the specimens tested survived 25 thermal cycles at which time only the surface laminae were semidetached; some of these surface laminae were slightly curled. A photograph of a typical specimen after testing is shown in figure 4. On the basis of macroscopic examination of the exterior of the specimens, using magnifications up to X40, it appeared that except for the surface laminae, the remaining laminae of the composite specimens were intact (bonded). Gaps were seen along the perimeter in these, as well as, other hot pressed specimens.

Modification II-A specimens. - The matrices in the modification II-A specimens had a grid pattern impressed upon their breadth surfaces as shown in figure 1(b). The surface laminae were 0.051 cm thick. The fact that the matrices were now continuous suggested that an improved intralaminar shear strength might be expected compared to the specimens with drilled matrices. Two modification II-A specimens underwent 50 thermal cycles each (table II) at which time the surface laminae were semidetached. The remaining laminae appeared intact on the basis of macroscopic examination at magnifications up to X40. Macrophotographs of a typical specimen after 50 fast cool thermal cycles are shown in figures 5 and 6; the latter figure indicates that the Nichrome surface laminae became detached because of intralaminar delamination of the adjacent tungsten laminae.

Modification II-B specimens. - These specimens had gridded interior matrix laminae and had surface laminae which were smooth and 0.003 cm thick. Two of these specimens were given 50 thermal cycles and survived without evident delamination damage (table II). All the laminae in one specimen appeared intact. A second specimen retained the separation that was present after initial hot pressing; this separation extended longitudinally inwards for about 0.5 cm from an edge. The improved resistance to delamination for modification II-B specimens may be explained by the following. When the surface matrix laminae cool, they contract. The inner side of a surface lamina is restrained from contracting by the adjacent tungsten lamina; hence a bending stress is generated by the outer lamina. The thicker the outer lamina, the greater the total force that must be resisted at the interface, up to the point where plastic deformation of the matrix may occur. Thus, the modification II-B specimens having the thin, 0.003 cm, surface lamina had a lesser tendency for surface layer delamination that the modification II-A specimens with thick, 0.051 cm, surface laminae. A photograph of a modification II-B specimen after 50 fast cool cycles is shown in figure 7.

Standard I-B specimens. - Two specimens of this configuration were thermally cycled (table II). One specimen was given 15 cycles and the other 21 cycles. Delamination began to occur in each specimen after two cycles. In both cases, delamination was progressive with cyclic testing and became extensive at cessation of testing as shown in figure 8.

Slow Cool Cycle Tests

Standard I-A specimens. - Ten standard I-A specimens were initially made, but only five of these specimens were found to be intact after cool down in the hot press. Thus, in a sense, five standard I-A specimens failed in 1/2 cycle. These specimens are not included in the tabulation of tested specimens. These failed specimens exhibited both interlaminar and intralaminar separations both of which involved most laminae. Much of this damage was visible to the unaided eye and other damage was visible only at the highest macromagnification used, that is, X40.

None of the modification I-A and II-A specimens having 0.051 cm thick surface laminae had any discernable delamination after slow cool down from the hot pressing temperature. This is in contradistinction to the fairly extensive damage noted for the standard I-A specimens.

One standard I-A specimen which successfully survived cool-down after hot-pressing was thermally cycled in a slow air cool test. It was given 11 heat-cool cycles at which time it was grossly damaged (table II).

Modification I-A specimen. - One specimen of this configuration was thermally cycled 25 times in vacuum (table II) at which time the surface laminae were curled and semidetached, while the remaining laminae were intact (fig. 9).

Modification II-A specimen. - One specimen of this configuration was slow cool thermally cycles 25 times in the vacuum hot press (table II) at which time the surface laminae were curled and detached while the remaining laminae appeared intact (fig. 10).

Modification II-B specimens. - Thermal cycling of two modification II-B specimens, 50 times in vacuum (in the vacuum hot press) produced no indication of delamination damage, table II and figure 11. One modification II-B specimen had five matrix and four reinforcement laminae; the other specimen had been previously given 50 fast cool thermal cycles and had the initial 0.5 cm long separation. The modification II-B specimen design showed resistance to delamination in both fast and slow cool thermal cyclic tests.

Standard I-B specimen. - Thermal cycling of a standard I-B specimen 50 times in the slow cool test (vacuum hot press) produced no indication of delamination, table II and figure 12. Comparison of the results obtained with the standard I-A specimens, which had the thicker surface matrix laminae, and the results obtained with the standard I-B specimen, which had the thin surface matrix laminae, in the slow cool thermal cycle test, suggests that the thicker surface laminae generated bending moments which caused many of the standard I-A specimens to extensively delaminate.

GENERAL REMARKS

As already mentioned, the maximum number of thermal cycles applied to any given specimen was 50. Since the modified specimens were intact after as many as 50 fast cool thermal cycles, and the standard specimens were essentially destroyed after 21 fast cool cycles, it would appear that the matrix modifications used had promise for significantly improved laminar composite thermal cycle failure resistance.

The observation that a laminar specimen withstood 50 thermal cycles would be more meaningful in th analysis of the results if this value could be directly compared with thermal cycle results obtained for monolithic materials. However, there were no comparative materials tested in this study. Also, reported thermal cycle test results on other materials involve the use of different testing procedures. While data for such a direct comparison were not obtained, a subjective appraisal of the results obtained in this study was made by comparing the 50 thermal cycles applied herein with the results for a number of monolithic materials tested in a pebble bed apparatus (ref. 10). From the reference study it was found that failure was observed in as few as 13 thermal cycles for some high temperature alloys. It is thus apparent that relatively few thermal cycles can cause failure of typical high temperature alloys in thermal fatigue tests. Further, it is believed that reasonably high stresses and strains were generated in the present study. Hence, it can reasonably be concluded that the 50 thermal cycle criterion used in this investigation is an indicator that laminar composites, with proper internal surface configuration, can have more resistance to thermal cycle failure than smooth internal surface laminar composites. The introduction of internal surface discontinuities along with use of thin surface matrix laminae appears to be a potentially viable way to improve the thermal expansion mismatch damage resistance of laminar composites.

SUMMARY OF RESULTS

The following major results were obtained from a study of the effects of discontinuities resulting from perforations through or grid patterned grooves on the surface of matrix laminae in laminar composites materials on their resistance of delamination. The composites studied contained tungsten reinforcement laminae and Nichrome matrix laminae and were either fast cooled or slow air or furnace cooled from 981^{o} C to room temperature.

1. The presence of matrix discontinuities improved thermal expansion mismatch resistance in specimens subjected to fast cool thermal cycles. Standard specimens (without matrix discontinuities) began to delaminate after 2 cycles and were virtually destroyed by delamination in 21 or fewer cycles. Some modified specimens (with matrix discontinuities) withstood 50 cycles, the maximum number applied, with no sign of delamination damage.

2. Modified specimens (with grid pattern grooves) with uniform lamina thickness (0.051 cm) showed delamination which was limited to the surface laminae while the rest of the specimens were intact (bonded) after the maximum of 50 cycles was applied.

3. Modified specimens (with grooves) and thin nongridded surface laminae (0.003 cm) survived the maximum of 50 fast cool or 50 slow cool cycles with no evident delamination damage.

4. Standard specimens with uniform lamina thickness (0.051 cm) delaminated severely in 21 or fewer cycles under both slow cool and fast cool conditions.

5. Standard specimens with thin surface laminae (0.003 cm) did not delaminate in 50 slow cool thermal cycles but delaminated in 21 or fewer fast cool thermal cycles.

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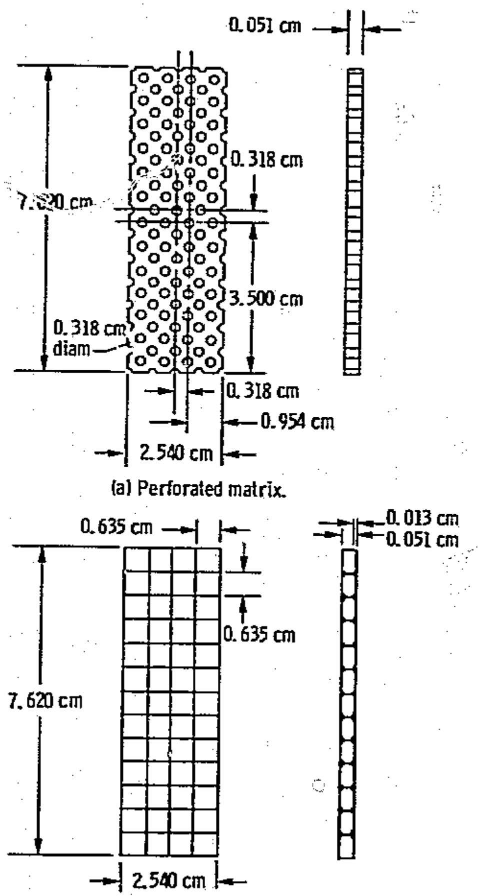
TABLE I. - DESCRIPTION OF SPECIMENS USED

Specimen designation	Surface condition of matrix laminae	Matrix laminae		Reinforcement laminae		Remarks
		Number	Thickness, cm	Number	Thickness, cm	
Standard I-A	Smooth	6	0.051	5	0.051	
Standard I-B	Smooth	6 (see remarks)	0.051	5	0.051	Surface laminae were 0.003 cm thick
Modification I-A	Perforated	6	0.051	5	0.051	
Modification II-A	0.635 by 0.635 cm grid pattern impressed on breadth surfaces	6	0.051	5	0.051	
Modification II-B	0.635 by 0.635 cm grid pattern impressed on breadth surfaces	6 (see remarks	0.051	5	0.051	Surface laminae were 0.003 cm thick. One specimen had 5 matrix and 4 reinforcement laminae.

TABLE II. - THERMAL CYCLE TEST RESULTS

Specimen designation	Surface condition of matrix laminae	Total number of specimens	cooling cycle	number of specimens	Maximum number of cycles	Remarks[a]
Standard I-A[b]	Smooth	5	Fast	4	21	Gross delamination in 21 cycles or less
			Slow	[c]1	11	Gross delamination in 11 cycles
Modification I-A	Perforated	6	Fast	5	25	Surface laminae were semidetached; rest of specimens intact (bonded). Some surface laminae were curled.
			Slow	[d]1	25	Surface laminae curled and were semidetached. Rest of specimen intact.
Modification II-A	0.635 by 0.635 cm grid pattern impressed on breadth surfaces	3	Fast	2	50	Surface laminae were semidetached; rest of specimens intact.
			Slow	[d]1	25	Surface laminae curled and were detached.
Modification II-B	0.635 by 0.635 cm grid pattern impressed on breadth surfaces. Surface laminae were smooth.	4	Fast	2	50	Stable separation 0.5 cm long in one specimen which was otherwise intact.[e] Other specimen intact.
			Slow	[d]1 [d,e,f]1	50	Specimens intact
Standard I-B	Smooth	3	Fast	2	21	Extensive delamination
			Slow	[d]1	50	Specimen intact

[a]Specimen evaluated macroscopically up to X40.
[b]Ten specimens were hot pressed but five of these delaminated extensively after cool down from hot pressing.
[c]Cooled in air.
[d]Cooled in vacuum.
[e]Cracks observed at free edges of grid indentations and generally appeared to extend inwardly for a distance corresponding to the width of the initial unbonded zone about the perimeter.
[f]Fifty fast cool thermal cycles prior to fifty slow cool thermal cycles.



(a) Perforated matrix.

(b) Gridded matrix.

Figure 1. - Schematic illustration of perforated and gridded matrix laminae.

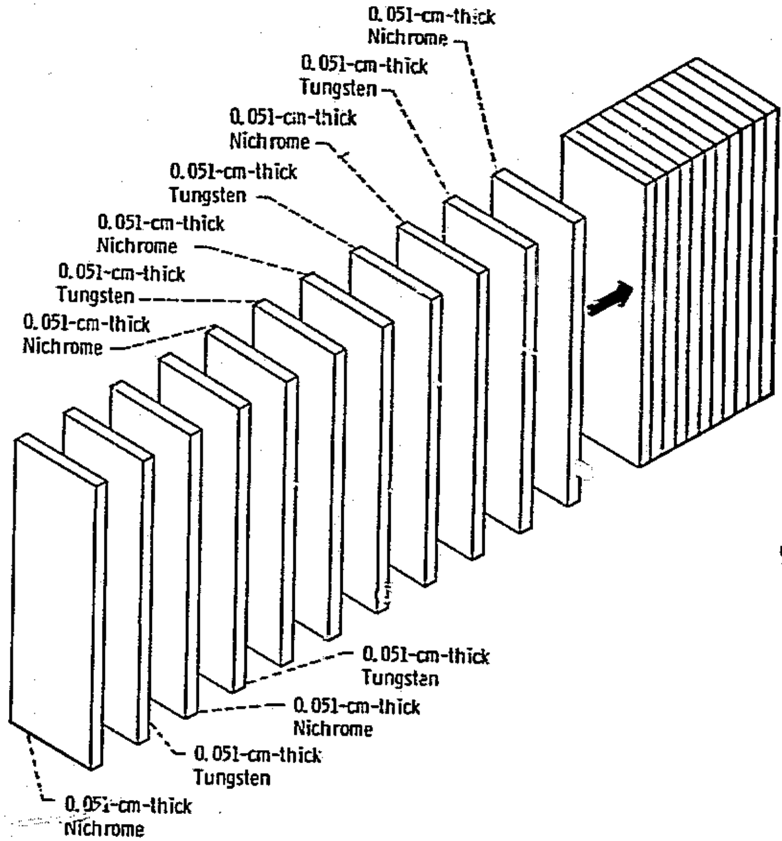


Figure 2(a). - Schematic representation of standard I-A specimen. Uniformly 0.051-cm-thick laminae, smooth matrix laminae surfaces.

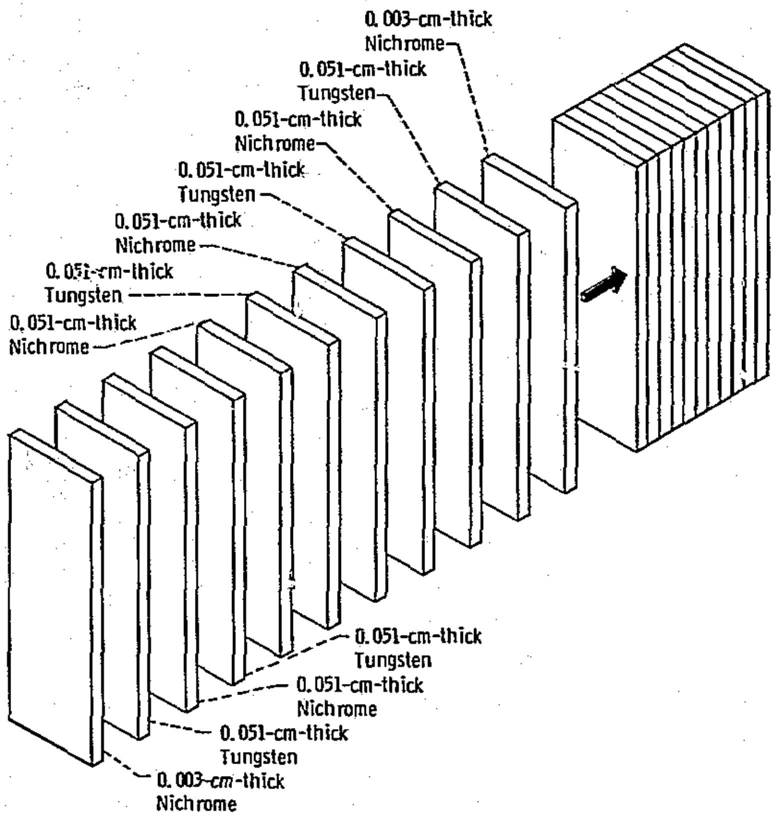


Figure 2(b). - Schematic representation of standard I-B specimen. 0. 003-cm-thick surface laminae, 0. 051-cm-thick interior laminae, smooth matrix laminae surfaces.

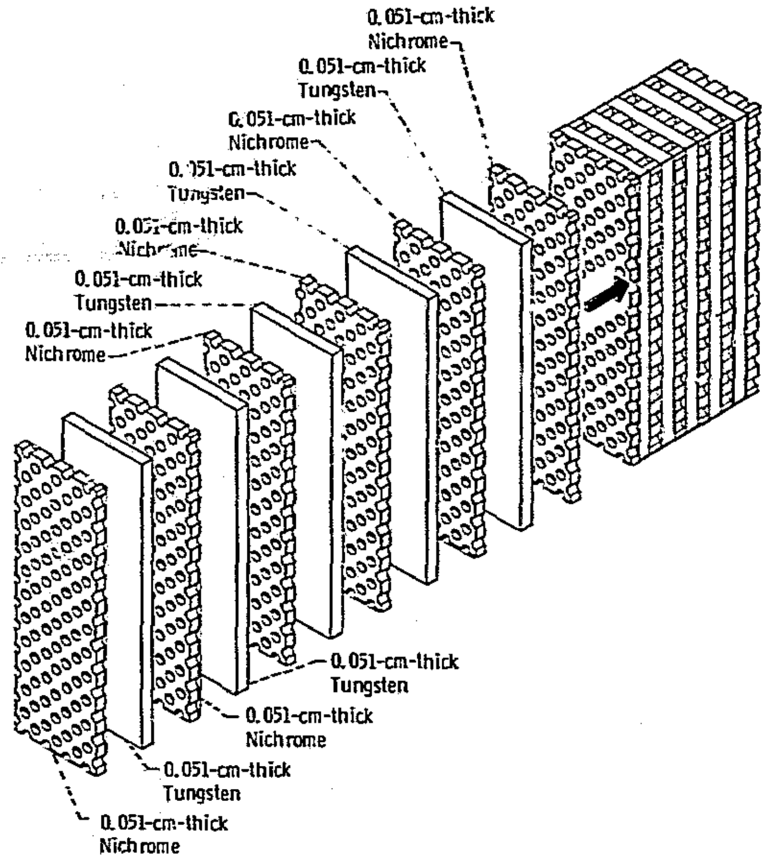


Figure 2(c). - Schematic illustration of modification I-A specimen. Uniformly 0. 051-cm-thick laminae, perforated matrix laminae.

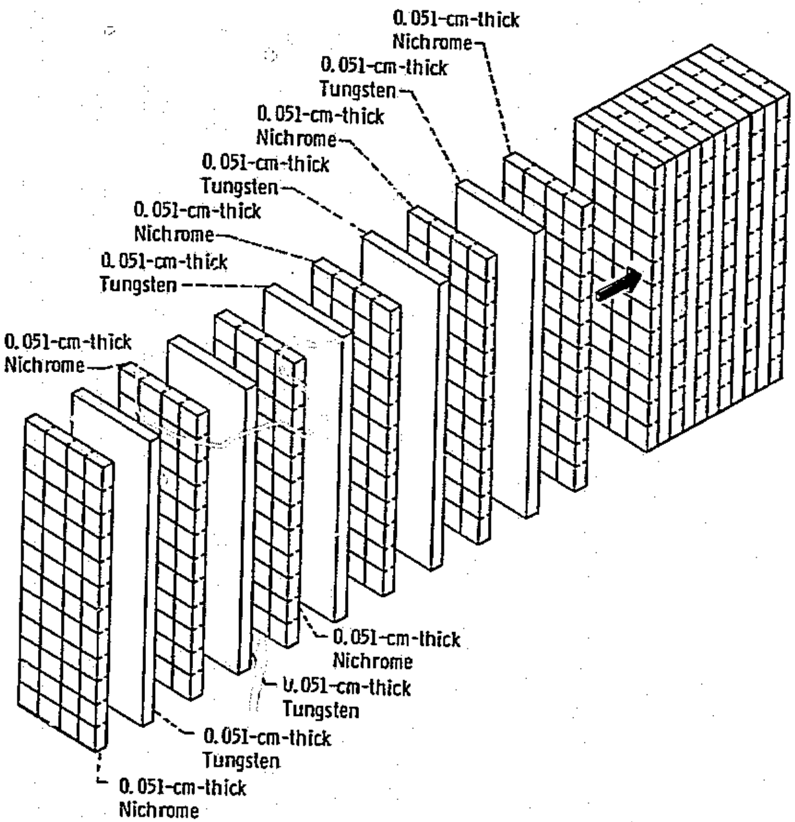


Figure 2(d). - Schematic illustration of modification II-A specimen. Uniformly 0.051-cm-thick laminae, gridded matrix laminae.

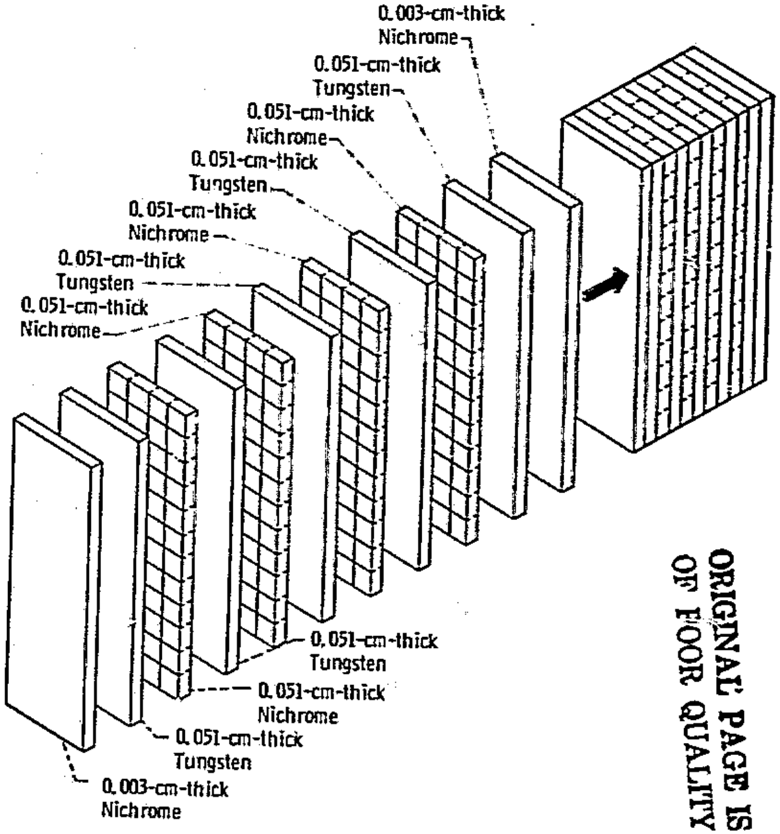


Figure 2(e). - Schematic illustration of modification II-B specimen. 0.003-cm-thick nongridded surface laminae, 0.051-cm-thick interior laminae, gridded interior matrix laminae.

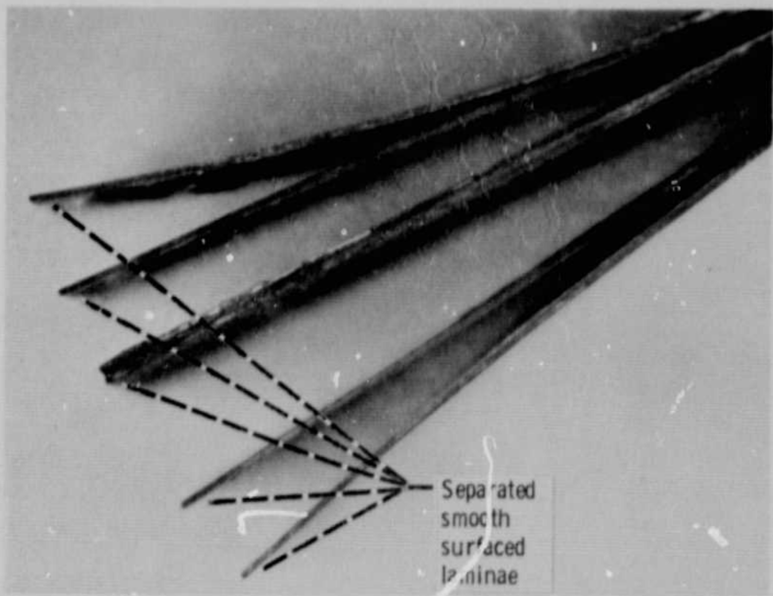


Figure 3. - Standard I-A Specimen after 21 Fast Cool Thermal Cycles.X2.

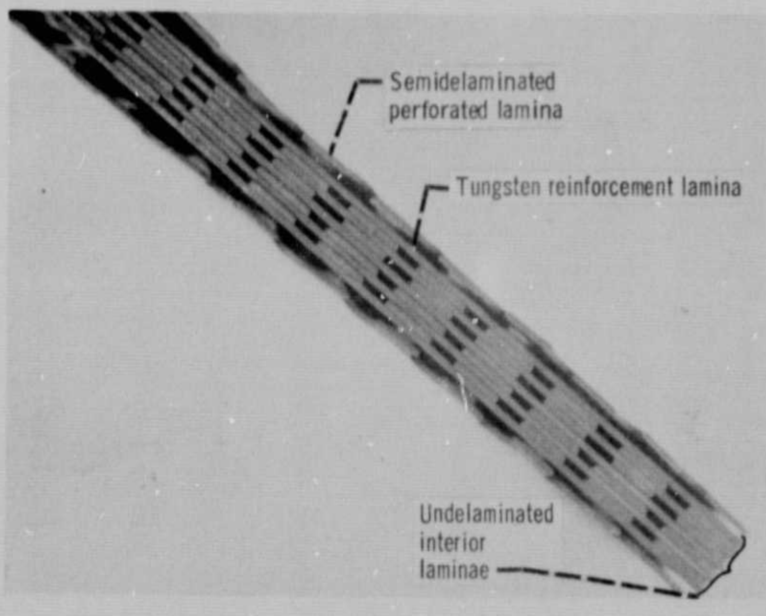


Figure 4. - Typical Modification I-A Specimen after 25 Fast Cool Thermal Cycles.XI.16.

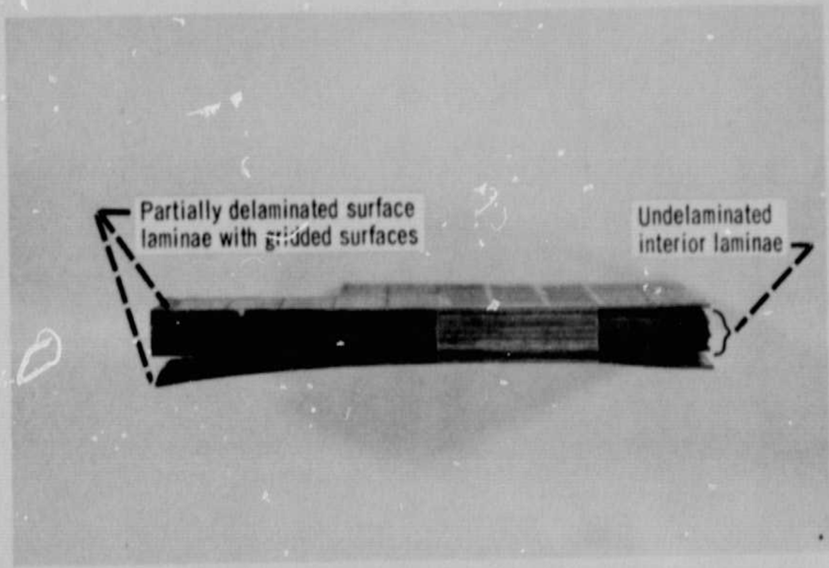


Figure 5. - Typical Modification II-A Specimen after 50 Fast Cool Thermal Cycles. X1.16.

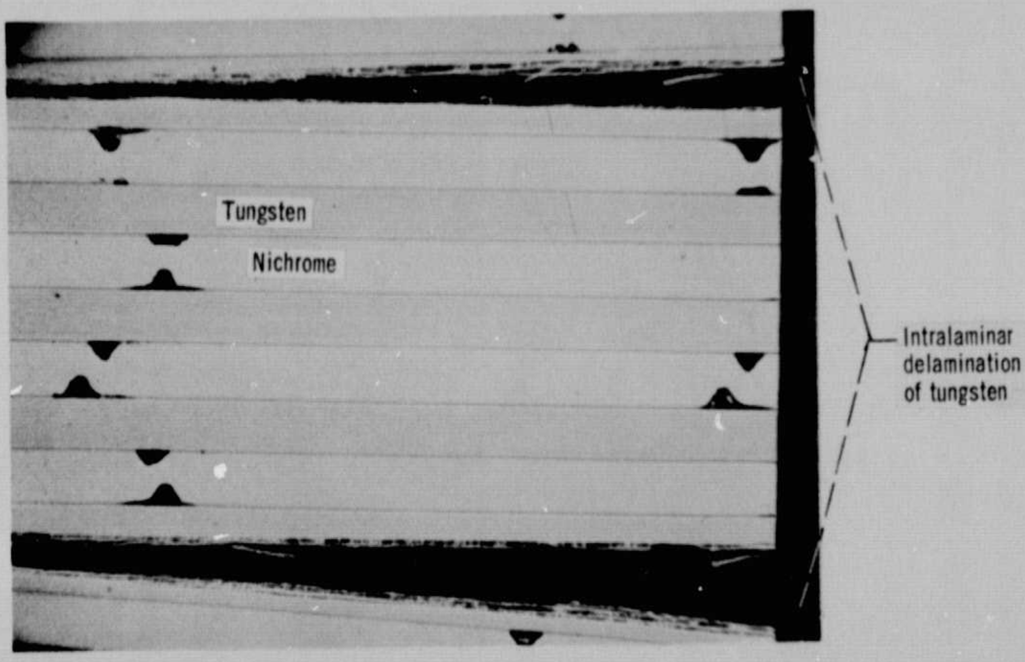


Figure 6. - Interior of Modification II-A Specimen after 50 Fast Cool Thermal Cycles showing Intralaminar Delamination of Tungsten. X16.

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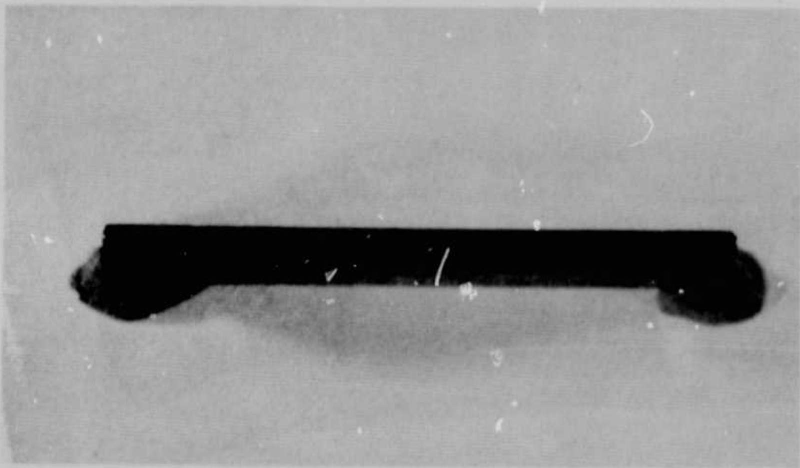

Figure 7. - Modification II-B Specimen after 50 Fast Cool Thermal Cycles. XI.16.

Figure 8. - Standard I-B Specimen after 21 Fast Cool Thermal Cycles. XI.16.

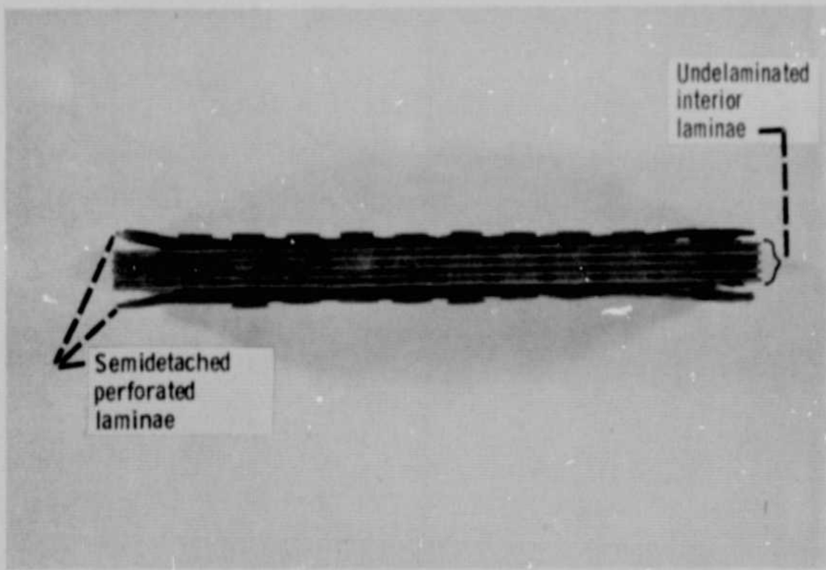

Figure 9. - Modification I-A Specimen after 25 Slow Cool Thermal Cycles. XI.16.

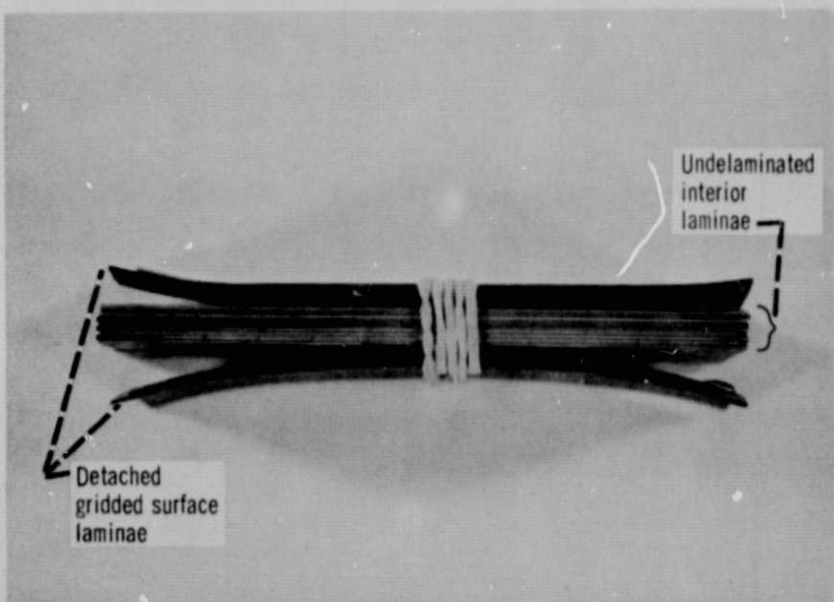

Figure 10. - Modification II-A Specimen after 25 Slow Cool Thermal Cycles. XI.16.

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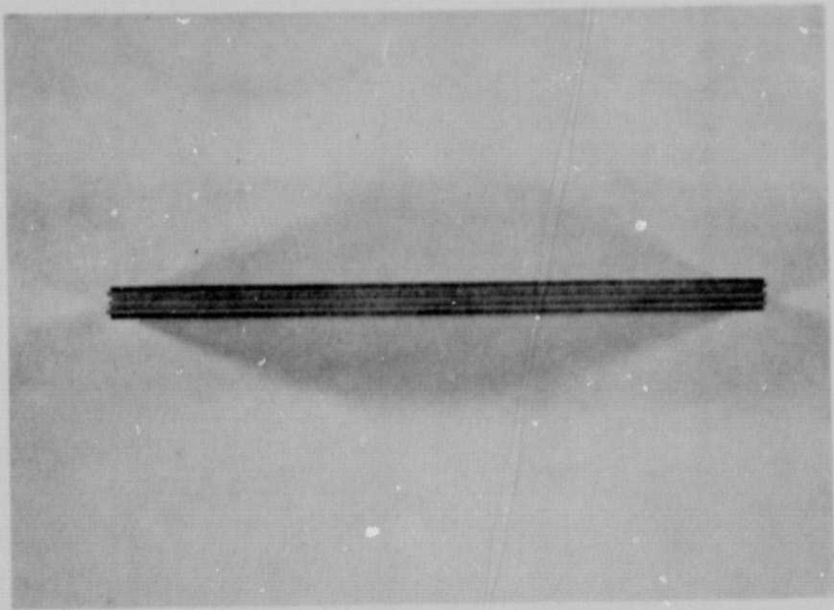

Figure 11. - Modification II-B Specimen after 50 Slow Cool Thermal Cycles. XI.16.

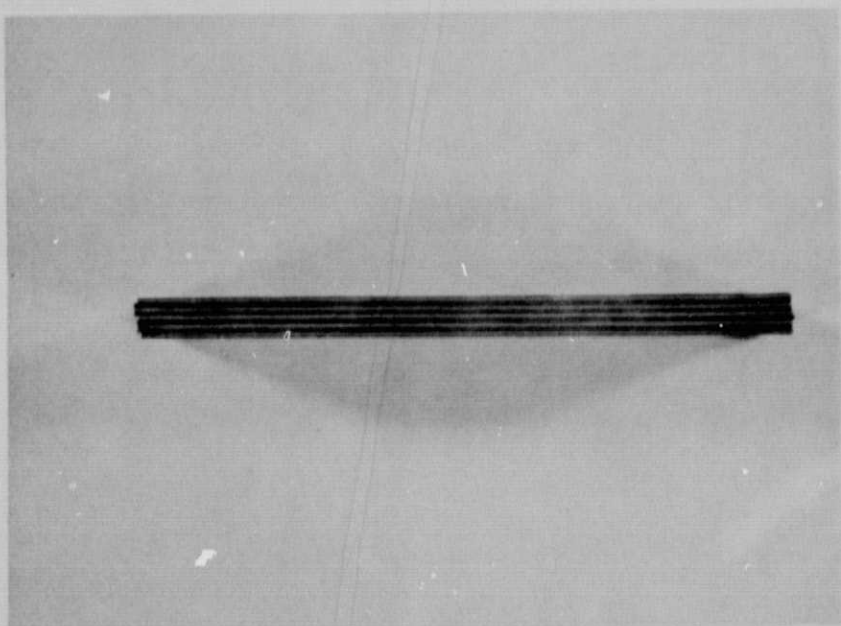

Figure 12. - Standard I-B Specimen after 50 Slow Cool Thermal Cycles. XI.16.