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THESIS

DETERMINATION OF THE DUCTILE TO BRITTLE TRANSITION TEMPERATURE OF PLATINUM-ALUMINIDE GAS TURBINE BLADE COATINGS

by

David J. Vogel

September 1985

Thesis Advisor: D. H. Boone

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Determination of the Ductile to Brittle Transition Temperature of Platinum-Aluminide Gas Turbine Blade Coatings

by

David J. Vogel
Lieutenant Commander, United States Navy
B.S., U.S. Naval Academy, 1974

Submitted in partial fulfillment of the
requirements for the degree of

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I. INTRODUCTION AND BACKGROUND

The development of the gas turbine engine has only excelled since the early 1940's, even though the principles for its operation have been known since the turn of the century. This is due to the simple fact that materials available before the 1940's could not withstand the high temperatures and stresses developed in the power turbine. Once the "superalloys" of iron, cobalt, and nickel-base with their solid solution strengthening and precipitation hardenability were discovered, gas turbine aircraft were developed for continuous high power operation in a relatively corrosion free environment.

The present use of gas turbines in combatant ships brings many advantages to the mariner such as quick starts, maneuverability, the ability to burn several different grades of fuel efficiently, and a greater horsepower to weight ratio than conventional power plants. However, the LM2500 engine developed for the U.S. Navy has encountered disadvantages in the marine environment in that relatively low power operation and high corrosive potential have led to earlier engine failure at the outset of the program than that experienced with gas turbine aircraft. While the superalloys solved the problems of high temperature and high

stress resistance which blocked the initial gas turbine development for aircraft use, the use of turbine blade coatings has alleviated to a significant extent the latter day problems encountered in the marine environment, although much work still remains to be done. The working out of compatibility problems between blade alloys and coatings in an effort to find the most corrosion resistant and economical combination will be a long process and may prompt one to ask, "why not develop a superalloy which can resist the environment on its own and forget about coating the blades?" This has been tried with some success but there is still no affordable superalloy which can form the required impervious oxide layer without excessive depletion of its constituent elements in the required high temperature, stress, and corrosion environment [Ref. 1, 2]. A superalloy blade with a coating designed to protect the blade from corrosion without degrading the mechanical properties of the superalloy has been the best long-term solution to the problem to date. At this stage the industry seeks to make blade-coating systems which will last longer under potentially worsening conditions.

Both nickel and cobalt base superalloys when used as airfoil substrates can achieve excellent mechanical properties including resistance to thermal and mechanical fatigue, high temperature creep lives, and high tensile strength. Even though the blades are coated in order to

enhance their resistance to degradation, they still must be resistant to oxidation and hot corrosion on their own in the event of coating compromise. Aluminum and chromium both can form protective oxides (Al_2O_3 and Cr_2O_3, respectively) and their presence in the superalloy in general enhances both oxidation and hot corrosion resistance. The choice of a superalloy for a particular component is usually dictated by the temperature and stress conditions in the particular section of the turbine under consideration. Cobalt-base superalloys are usually more corrosion resistant than nickel-base superalloys, due in part to their high chromium content, and thus they are used for vanes. Nickel-base superalloys on the other hand have lower melting points yet much greater strength than the cobalt-base superalloys and are used for blades as well as some later stage vanes.

A. GAS TURBINE BLADE COATINGS

To select the proper coating for a given substrate in a particular application, six basic requirements must be met. The coating must:

1. be highly resistant to oxidation and hot corrosion;
2. have the ductility during start-up, power transients, shut-down, and at temperature to accommodate substrate dimensional changes without allowing crack initiation;
3. be compatible with the substrate superalloy in terms of both thermal expansion and elemental constitution;
4. not interdiffuse to too great an extent with the substrate superalloy such that mechanical properties of the substrate will be degraded;

5. be easily applied to the substrate superalloy; and

6. have a low cost in relation to life improvement. [Ref. 3]

The industry's best solution to the problem to date has been in the form of aluminide diffusion coatings and metallic overlay coatings.

Metallic overlay coatings, the newest of the two types and commonly referred to as "MCrAlY" alloy coatings (M=Fe, Ni, and/or Co), are applied by either physical vapor deposition processes (PVD), as typified by sputtering and electron-beam evaporation, or most recently by low pressure chamber spray techniques [Ref. 4]. Structurally, the coatings consist of two phases--an aluminide phase which is brittle dispersed in a chromium-rich solid solution matrix which is ductile. The chromium and aluminum are present to form the protective oxide layers mentioned earlier and the yttrium and/or other active elements ensure that the oxide layers adhere to the coating surface [Ref. 5]. Because metallic overlay coatings depend very little upon substrate element incorporation, they degrade the substrate mechanical properties to much less a degree than do aluminide diffusion coatings. However, as a result of high processing costs and problems encountered in maintaining tight compositional control with the PVD methods, aluminide diffusion coatings are generally regarded as the most advantageous overall, taking into account the six established requirements for coatings [Ref. 6].

Aluminide diffusion coatings are formed on the substrate by a relatively simple and inexpensive process called pack cementation which is a gaseous chemical vapor deposition and diffusion process conducted in an inert 'pack' mix. The resulting coating consists of a zone which lies directly on top of the substrate made up of an intermetallic compound (usually β(NiAl) for nickel-base superalloy substrates) plus one or two more outer zones also consisting of intermetallic compounds [Ref. 7]. An environmental barrier is formed by the oxidation of aluminum (Al_2O_3) and the surface is continuously fed additional aluminum for oxidation in the event scale damage occurs due to thermal fatigue (spalling) or erosion. Once the aluminum content of the coating falls below the critical level for the Al_2O_3 scale to form, substrate element consumption takes place and the coating must be replaced.

In practice it is possible for only two types of aluminide diffusion coatings to form. The product formed will depend upon the activity of the aluminum and the formation temperature. A high activity pack with a relatively low formation temperature will produce what is called an "inward" aluminum diffusion coating because aluminum diffuses "in" followed by outward nickel diffusion during subsequent diffusion treatment and a three zone structure of β(NiAl) and precipitated substrate elements

forms. Alternatively, if a relatively high formation temperature with a low aluminum activity is chosen, an "outward" nickel diffusion will occur in the β(NiAl) and a two zone structure will result showing a nickel-rich β(NiAl) phase on the outside with a β(NiAl)/substrate phase mixture next to the substrate. Inward and outward aluminide coatings are shown in Figures 1 and 2.

B. THE INCORPORATION OF PLATINUM INTO THE DIFFUSION COATING

The two devastating enemies of the gas turbine blade are oxidatiton and hot corrosion. The diffusion aluminide coatings described above were first developed and used with excellent results in providing oxidation resistant superalloy coatings for alloys such as Udimet 700 and B-1900 first stage gas turbine blades in aircraft [Ref. 8]. As the diffusion aluminide coating became more widely used, it established itself as an excellent life extender for most superalloy blade materials. However, as lower and lower grade fuels came into use and the coatings were used more extensively over a wider temperature range in the marine environment, hot corrosion attack became the major cause of coating and blade failure. Hot corrosion is defined as the combined attack of high temperature combustion exhaust gases and sea salts in condensed phases on the surface of a turbine component. Thus, hot corrosion is experienced predominantly in the marine environment since it requires

the presence of sodium and sulfur in the form of sodium sulfate. The sulfur is provided by low grade high sulfur fuel, and in a given temperature range, sodium sulfate condenses on the turbine blades and components. The blade's protective oxide can be dissolved by the fluxing action of the molten salts. Once the protective oxide is gone and unable to reform and the substrate is exposed, the hot exhaust gases cause further accelerated oxidation of the unprotected metal and eventual loss of mechanical strength and component integrity [Ref. 9]. Further, it has been determined that two distinctively different types of hot corrosion can occur. Basic fluxing (TYPE I) hot corrosion is associated with temperatures in the range 800C - 950C. This was the first type of hot corrosion noted in high power level aircraft operating in the marine environment and it is characterized by the presence of sulfides embedded in the broken-up and degraded metal surface. However, propulsion units operating at lower power levels, and thus developing lower airfoil surface operating temperatures, experienced a different and potentially more devastating type of hot corrosion in the range 675C - 775C. This acid fluxing (TYPE II) hot corrosion results in a relatively smooth surface appearance with many pitting-type corrosion fronts causing the metal surface to rapidly recede [Ref. 10].

Experimentation in the industry led to the surprising discovery that in some cases the incorporation of the noble

metals such as platinum into the conventional diffusion aluminide coating could greatly enhance the oxidation and hot corrosion resistance of the conventional aluminide coating-substrate systems already in use. The first commercial "platinum-aluminide" coating was developed by Lehnert and Meinhardt in Germany in the early 1970's and was licensed by the Howmet Corporation as LDC-2 and by Chromalloy as RT 22. It improved blade life in cyclic oxidation at least fourfold and resistance to at least some types of hot corrosion twofold over conventional aluminide diffusion coatings [Ref. 11].

To construct the platinum-aluminide the conventional aluminide coating was modified by first electrodepositing 10 μm of platinum onto the bare nickel-base superalloy substrate before the normal aluminizing of the blade at high temperatures [Ref. 12]. While many studies have confirmed that the reason for the increased substrate protection is the enhanced aluminum oxide scale adherence caused by the platinum, the actual mechanism for this phenomenon is not fully known [Ref. 13]. It was originally thought that platinum would be incorporated in the interdiffusion zone where it would tie up the aluminum sufficiently during thermal cycling of the turbine blade to moderate aluminum diffusion to the surface, thus extending the life of the coating by allowing the aluminum that was present to be used

over a longer period of time for oxide scale replacement. This "diffusion barrier" theory was quickly dispelled, however, by microprobe analysis which revealed that on the LDC-2 coatings as well as on other platinum-aluminides the majority of platinum is at the very outside (surface) region of the coating rather than in the interdiffusion zone or diffused into the substrate [Refs. 14, 15].

At the U.S. Naval Postgraduate School in Monterey, California, Boone and Deb have studied the wide range of variables possible for forming the platinum-aluminide coatings and determined that platinum deposition method, pre-aluminizing heat treatment, and subsequent aluminizing treatment most affect the final structure and hot corrosion resistance of the platinum-aluminide coating formed while the pre-aluminizing heat treatment appears to be the dominant factor in determining the coating structure [Ref. 16]. This is an important observation, because while commercially available platinum-aluminide coatings have been in use for years including the LDC-2, Johnson-Mathey's JML-1, and Chromalloy's RT-22, the reasons for their success, other than the fact that "they work," have been hard to establish due to the proprietary nature of manufacturing processes and test procedures.

Depending upon the pre-aluminizing heat treatment used, the platinum-aluminide coating structure can range from a

continuous $PtAl_2$ surface on top of a thinly interdiffused platinum zone to a two phase $PtAl_2$ + NiAl (Pt in solution) structure to a single phase NiAl(Pt) coating system. Keep in mind, however, that the platinum deposition process has an effect on the formation of the coating and the aluminizing treatment also affects these structures apparently causing in an analogous manner the "inward" and "outward" diffusion noted earlier in the conventional aluminide coatings.

Boone and Deb [Ref. 17] reported improved but varying resistance to TYPE I hot corrosion as a function of coating morphology and other researchers have delineated the virtues of platinum-aluminide coatings in resisting high temperature degradation. However, a great many questions remain regarding the mechanical properties of platinum-aluminides [Refs. 18, 19, 20]. It is significant that Boone and Deb reported their results based on coating morphology due to processing treatments.

C. THE COATING SYSTEM DUCTILE TO BRITTLE TRANSITION TEMPERATURE (DBTT)

Throughout the literature platinum-aluminides are generally regarded as "brittle" in comparison to other pack cementation and overlay coatings, but no detailed test results are presented to identify which platinum-aluminide coatings are more or less brittle than others of their

genre. Indeed, mechanical properties tend to be considered in the wake of advancements in the protective nature of the coating itself. However, mechanical properties of the coating systems are also important and can significantly influence coating-substrate lives. High and low cycle fatigue, thermal fatigue, crack growth, creep, rupture, and tensile properties as well as coating ductility are vitally important to the coating-substrate system and engine design. If any of these coating properties are insufficient or incompatible with the substrate they protect, coating penetration results and the substrate is degraded by its environment at an accelerated rate.

Because coatings and substrates always display a phase and compositional difference from one layer to the next due to the diffusional nature of the platinum-aluminide coatings and the tightly bonded intermetallics which form, complete compatibility of mechanical properties at all temperatures is an impossibility. It has been noted that since most coatings consist of an intermetallic phase(s) with a BCC structure, they behave in a brittle manner at low temperatures which changes to a more ductile behavior at higher temperatures [Ref. 21]. The level of brittleness and the temperature of transition from brittle to ductile behavior are strong functions of intermetallic phase composition and stoichiometry. This in turn is dependent

upon the coating-substrate composition and processing procedure. Because of this, coating-substrate "systems" are studied rather than separating the two for examination and the ductile to brittle transition temperature (DBTT) is considered one vital indicator of system performance.

The coating-substrate system behavior is a strong function of thermal expansion mismatches and resulting residual stresses between the two as well as a function of differing mechanical properties between coating and substrate which can cause additional strain to occur in the coating. The coating's ability to accommodate this strain is its ductility. One can clearly see the vital nature of temperature related ductility data--the procedures for start-up and shut-down of a gas turbine engine as well as the blade design and cooling scheme must take into account the coating-substrate ductility limits at all temperatures from start-up through the entire operating range to ensure that thermally and mechanically induced strains do not exceed those which can be accommodated at any given temperature. Turbine start-up and no-load warm-up times as well as operating and shut-down procedures must accommodate this data to ensure the coating is not compromised.

Lowrie [Ref. 22] was the first to realize the importance of the DBTT, and in 1952 he established a relationship between the melting temperature of a number of aluminide phases and the DBTT:

$$DBTT = 0.6 \text{ to } 0.7\ T_m(K)$$

Strang and Lang used this relationship to project that the DBTT range of $PtAl_2$ (870°C - 1070°C) is slightly higher than that of NiAl (868°C - 1060°C) and much higher than those of two other intermetallic compounds which are found in the conventional aluminide coatings on a nickel-base superalloy substrate, Ni_3Al (730°C - 900°C) and Ni_2Al_3 (570°C - 710°C) [Ref. 23]. This might indicate that the platinum-aluminide is more brittle since it remains in the brittle mode through a greater temperature range from room temperature through start-up, and indeed a high DBTT is an undesirable characteristic. A low DBTT would ideally place the coating-substrate system into a ductile mode by the time turbine warm-up is complete and before heavy stress is placed on the engine.

An additional consideration is that even though most coatings are brittle at temperatures below 700°C, they must be able to withstand thermally induced strains which are characteristically largest at the airfoil surface. As Strangman and Boone point out these strains are on the order of 0.15 to 0.35 percent and a coating which cannot maintain this much ductility at the low temperatures will not be adequate [Ref. 24].

But again, this thought about platinum-aluminides is merely a "projection" of Lowrie's original findings and is

not a result of actual laboratory testing. Furthermore, it applies to the $PtAl_2$ phases specifically, and while $PtAl_2$ is a continuous phase at the surface in some platinum-aluminides, it exists as a second phase with NiAl(Pt) in others. Without actual testing, the DBTT of a specific coating substrate system cannot be ascertained or discussed with any reliability.

While the temperature range in which the system is in transition from brittle to ductile behavior is often used as above, the industry standard definition of DBTT was set forth by Lowrie and Boone in 1977 as that temperature which corresponds to a fracture strain of Ø.6% [Ref. 25]. At temperatures above the DBTT, strains greater than Ø.6% can be accommodated and at temperatures below the DBTT strains up to Ø.6% can be accommodated. The most important point to observe in the definition is that testing is done to coating fracture so that the strains recorded in any experiment are actually beyond the maxima that can be tolerated at their given temperatures. In a typical DBTT tension test this involves coating fracture only at temperatures below the DBTT while at temperatures above the DBTT substrate failure may occur with or without coating fracture [Ref. 26]. In the brittle region cracks are straight and close together while in the ductile region cracks are more separated and bifurcated. See Figure 3.

Brandis and others [Ref. 27] have shown that the LDC-2 platinum-aluminide coatings on IN738 substrates show a slightly higher DBTT than conventional aluminide coatings, and others have drawn attention to the fact that platinum exists in solid solution in the NiAl phase of platinum-aluminide coatings and would likely effect the DBTT of platinum-aluminide coating-substrate systems [Refs. 28, 29].

In continuing the gas turbine blade coating research effort at the Naval Postgraduate School, it is the purpose of this thesis to develop and demonstrate a procedure using NPS equipment to determine coating system ductility and to specifically determine the ductile to brittle transition temperatures (DBTT) of representative platinum-aluminide coating-substrate morphologies for NPS standard platinum diffusion heat treatments and aluminizing heat treatments which are recognized throughout the industry. These results should establish data involving a critical coating mechanical property and supply needed design information for both the Navy and industrial manufacturers of turbine blades. Specifically, while the $PtAl_2$ compound is regarded as brittle, its contribution to or degradation of the ductility of representative platinum diffusion heat treatments and aluminizing activities and temperatures will be ascertained.

II. EXPERIMENTAL PROCEDURES

A. SPECIMEN PREPARATION

The substrate on which all the coatings were applied and tested was IN738, a commercial nickel-base superalloy with composition delineated in Table I. The IN738 tensile specimens were machined from the cast state to the dimensions shown in Figure 4, although careful measurement of the final gage length after the platinum-aluminide coating was applied was made for each specimen due to the critical dependence of strain on this dimension.

The final diameter was not necessarily consistent from one specimen to another due to the fact that some specimens had to be ground more to eliminate casting porosity for consistent coating adherence. Slightly differing gage diameter was not a factor, however, in this experiment since only strain was of importance and not the load picked up by the specimen to impart the strain.

All specimens were ground to a 12 micro-inch RMS surface finish for consistent coating surface structure. The specimens were processed at commercial facilities to develop the Pt-Al coating stucture desired for testing. Processing involved the electro-deposition of a 10 μm thick layer of platinum followed by a heat treatment to diffuse the

platinum into the substrate. Once this was accomplished, the specimens were subjected to an aluminizing treatment. There are two aluminizing processes adopted as the NPS standards. One (LTHA) is a high aluminum activity process performed at low temperature (700°C), and the other (HTLA) is a low aluminum process performed at high (HTLA) temperature (1025°C). Without the incorporation of platinum these processes respectively yield the inward and outward coatings described earlier. Details of the platinum-aluminide coating process and resulting structures are discussed in greater detail elsewhere [Ref. 30].

Table II describes the coatings selected for testing and Figures 5, 6, 7, 8, and 9 display the coating morphologies.

Four tensile specimens and one test pin per coating were processed so that at least four data points on the DBTT curve could be obtained for each coating. The test pin for each coating was used for metallographic purposes to ensure that each of the coatings was formed properly. Each of the photographs in Figures 5-9 were taken of portions of the test pins which had been mounted, polished, and etched with AG-21 (a diluted HNO_3 etchant applied by swabbing and used to develop contrast).

Because initial testing performed on the coated specimens revealed that the substrates were too brittle to

yield conclusive results, two additional heat treatments of solution and aging were performed on all of the remaining specimens. These treatments of 1120°C for two hours in an inert atmosphere and 845°C for twenty-four hours were used by Lehnert and Schmidt on IN738 substrates in previous DBTT experiments and proved effective in restoring substrate ductility so that testing would yield tangible results [Ref. 31].

The final coating structures (Figures 5-9) display the characteristic phases that are common to the platinum-aluminides. Coating Nos. 1, 3, and 5 (Figures 5, 7, and 9 respectively) display the two phase structures discussed in Chapter I composed of the $PtAl_2$ and NiAl (Pt in solution) phases, while coatings Nos. 2 and 4 display the single phase NiAl(Pt).

B. TEST SYSTEM DESIGN

The testing method devised to establish the DBTT of the platinum-aluminide coatings was a strain-to-fracture method specifically designed to use existing Naval Postgraduate School equipment, which includes the Model TT-D Instron Tensile Testing machine and the Model 2232 Marshall clamshell oven. In most DBTT testing the coating system is strained at the elevated temperature of interest, and the coating is either observed to fail visually if a windowed oven is used [Ref. 32], or an audio method is employed.

Another standard practice in industry is to heat a coated tensile test specimen to the test temperature and strain it a predetermined amount. Then the specimen is unloaded and cooled to room temperature. Plastic surface replicas are then taken to determine if any coating cracks have occurred. If they have not, the specimen is again heated to the test temperature and strained a greater amount. This more time consuming process continues until coating cracks are detected and thus a data point at that test temperature is achieved [Ref. 33].

In this instance, the audio approach was employed in which a sound transducer, the Dunegan model S-140B, was used in conjunction with the Tektronix Type 551 Dual Beam Oscilloscope to give a visual signature of the coating failure. This method was considered feasible due to evidence that coating cracking on the surface of a coated alloy yielded a distinct acoustic signature in toughness tests reported by Lehnert and Schmidt on platinum-aluminide coatings [Ref. 34]. Surrounding noise which includes gear noise in the Instron machine and even thermal expansion of the tensile specimen appears sinusoidally, and is easily distinguished from coating crack generated noise. See Figure 10. The sensing element was attached directly to the lower pullrod in order to pick up the transmission of the cracking sound as directly as possible. See Figure 11.

The extensometer, pullrods, and grips were designed first and foremost to accommodate temperatures in excess of 1000°C without deformation, therefore MAR-M246 pullrods and grips were employed as well as an extensometer made entirely of IN601. Secondly, the dimensions were calculated such that the test specimens could be held in the center of the oven, which has a 5 inch hot zone maintained by the three evenly spaced heating elements through the temperature range 500°C-850°C, and ample room for at least two insulation pads at the top and bottom of the oven would be provided. All of these dimensions were computed with the crossheads a full seven inches above its bottom position even though elongations of a maximum 0.02 inch were expected. See Figure 11.

The gage length of each coated specimen was measured to 0.001 inch, the standard being 1.400 inch. The measured portion was only the portion of consistent diametrical dimension (Figure 4) as specified by ASTM standards and the maximum runout on any one specimen was 0.005 inch through the entire gage length. In all testing cases cracking was observed around the entire sample indicating good specimen alignment and sufficient specimen trueness (no bends on gage length).

The extensometer was attached to the specimen 1/16 inch above and 1/16 inch below the square shoulders of the gage

portion and not on the gage length itself to prevent extensometer induced cracking. It was assumed that the elongation in areas of increasing thickness above and below the gage length but inside the extensometer grips would be insignificant compared to elongation on the gage length itself. The grooves on the specimens were cut at a 60 degree angle so that the 120 degree circular wedge grips on the extensometer would have a two surface grip as illustrated in Figure 4.

As each specimen was clamped onto the extensometer a Type "K" alumel-chromel wire thermocouple was affixed to read the temperature at the center of the specimen so that the actual specimen temperature could be checked on a digitial readout of $\pm 1^{o}C$ to corroborate the temperatures registered by the three calibrated furnace coils. One thermocouple was used throughout the testing and it was checked with ice bath and boiling water periodically to ensure accuracy.

A dial gage accurate to $\pm$0.0001 inch registered specimen elongation on the platform section of the extensometer external to the furnace (see Figure 11). The dial gage was maintained as the true indicator of specimen elongation since the Instron chart movement registers only crosshead movement, not necessarily equal to elongation, especially once the specimen commences to deform plastically.

The strain referred to in all results was computed as engineering strain to the nearest 0.01% using the equation:

$$\varepsilon(\%) = \frac{\Delta L}{L} \times 100$$

where "ΔL" is the elongation read from the dial gage to the nearest 0.0001 inch for the first occurrence of the coating crack signature in each test, and "L" is the tensile specimen gage lengh measured to 0.001 inch prior to testing and adjusted for any thermal expansion undergone prior to straining the specimen. For example, the test on coating No. 2 specimen No. 3 took place a 750°C. Cracks were noted on the oscilloscope first at an elongation of 0.0085 inch on the dial gage. The specimen's original gage length prior to testing was measured at 1.403 inch and it expanded 0.001 inch (noted on the strain gage) while heating to the test temperature. The strain at coating failure was thus computed to be:

$$\varepsilon(\%) = \frac{0.0085 \text{ inch}}{1.404 \text{ inch}} \times 100 = 0.605\% \simeq 0.61\%$$

See Table III.

C. SPECIMEN TESTING

Using the definition of the transition temperature to be that temperature at which 0.6% strain can be imparted before

coating failure, a transition temperature range was "guessed" for each coating based upon data available for similarly formed nickel-aluminide coatings without platinum. Since platinum-aluminide coatings are regarded as more brittle structures, and thus would have higher transition temperatures in general, 50°C was added to the "guess," the oven was clamped around the coated test specimen, and the specimen was heated to this adjusted temperature.

It was imperative during this heating and cooling stage to ensure that the lower pin attaching the crosshead to the lower pullrod was removed and that the crosshead was lowered to allow for expansion of the specimen and pullrods during heating so that a compressive load would not be imparted to the specimen or to the extensometer. Once the high guess temperature was reached, the lower pin was inserted and the no-load setting on the Instron chart was set. All specimens exhibited some thermal expansion due to this process which ranged from 0.001 to 0.004 inch, but the dial gage was zeroed prior to loading so that all testing was done isothermally--registering only that strain at temperature imparted by the Instron machine crosshead motion. The initial gage length was adjusted for thermal expansion as noted earlier.

After the desired temperature was stable for 10-15 minutes with no load imparted, the crosshead was engaged and

a load was applied to the specimen. A chart speed of 5 inches per minute and a crosshead speed of 0.01 inches per minute were used giving a magnification factor of 500. The strain rate was 0.007 per minute and this was maintained throughout the testing in order to achieve consistent results due to the dependence of the DBTT upon strain rate [Ref. 35]. Once 0.8% strain was imparted, if no cracking was observed on the oscilloscope, it would be assumed that the upswing of the DBTT curve had been located. The load would then be removed from the specimen and the lower pin removed. Then the furnace would be reduced in temperature by 50°C and the test would be resumed as described. If cracking resulted in this specimen and if it were in the ductile region (above 0.6% strain) the cracks would likely be few and far apart. This same specimen could be used later at room temperature to establish the low end of the brittle region, since cracking in this area is short and dense and easily distinguished from the cracking initated in the ductile region. See Figure 3.

At this point the second of four specimens per coating would be tested at a temperature 100°C lower, and the remaining two specimens would be used between and above the temperatures already located to fill in the high curvature portion of the graph. Thus a maximum of five data points on the DBTT curve could be achieved with the four tensile specimens bearing each coating.

D. ACCURACY

Test results are a function of two measurements, temperature and elongation, and both were recorded with calibrated instruments. These instruments were assumed to yield true readings within their indicated accuracies. Because concurrent devices were not used to record strain or temperature due to space considerations, a comparison of readings or an error analysis were not possible. Inaccuracies certainly exist, and also the data which appears in Figures 12 and 13 would certainly have some scatter if multiple specimens were used at each single temperature with multiple sensors. However, comparisons of successive data points revealed a high level of data consistency.

Results are a weak function of temperature as shown by the temperature dependence and little or no difference in ductility is expected with small variations of temperature. The error in engineering strain, however, can be calculated because a difference of $\pm$Ø.Ø1 inch of elongation in a typical case would yield an error in ε of $\pm$Ø.Ø1%. Error bars were not included on the graphical presentation of the results (Figures 12 and 13), however, because this kind of accuracy should not be indicated with so few specimens tested to construct the plots.

III. RESULTS AND DISCUSSION

A. TEST OBSERVATIONS

As discussed in Chapter II, the initial testing done on two specimens with coating No. 1, one specimen with coating No. 2, and two specimens with coating No. 5 resulted in brittle substrate failures, and while a very distinct signature was observed on the oscilloscope (see Figure 10a) prior to specimen fracture, no coating cracks were evident on the specimens. Post coating heat treatments discussed in Chapter II were employed to restore the mechanical properties (ductility) of the substrates on the remaining coated tensile specimens, and valid data points were achieved in all subsequent tests.

All test results are presented graphically in Figures 12 and 13, and the data points used to construct these graphs are displayed in Table III. The platinum-aluminide coatings formed with the LTHA process are shown in Figure 12 compared to data presented by Goward for the nickel-aluminide coating formed by the LTHA process. The platinum-aluminide coatings formed with the HTLA process are shown in Figure 13 compared to data presented by Goward for the nickel-aluminide coating formed by the HTLA process. [Ref. 36]

Once several specimens were tested, it became very clear what the signature of a coating crack looked like on the

oscilloscope (see Figure 10b). Whenever this sharply defined characteristic was exhibited on the oscilloscope, there were cracks which could be observed on the specimen gage length at 425x under an optical microscrope, and whenever this signature did not appear there were no cracks, regardless of how much strain the specimen had undergone and no matter what other patterns appeared on the oscilloscope. This is considered reasonable proof that the oscilloscope signature shown in Figure 10b was coating cracking observed during tensile straining of the specimens. Further confirmation of the cracking signature was provided by testing done above 800°C. The specimens deformed very quietly at these temperatures, they registered minimal noise on the oscilloscope, and so the cracking signature was even more distinguishable than at lower test temperatures where other noise was present on the scope. For instance, the test of coating No. 2 specimen No. 1 conducted at 860°C showed practically a clear scope, even though it was strained to 1.71%. Optical microscopy revealed no cracking of the specimen. Coating No. 3 specimen No. 1 on the other hand, which was also tested above 800°C, displayed the distinctive signature shown in Figure 10b beginning at 0.25% strain and continuing until approximately 0.65% strain with a clear scope showing beyond that point until the specimens fractured at 2.20% strain. Examination of this specimen

after the test revealed that the coating cracks on the gage length were clearly visible and characteristic of a brittle coating failure (see Table III and Figure 3).

Another lesson learned during this testing that was not initially evident was that some plastic deformation had to be imparted to each specimen after the coating cracked in order to open the cracks enough so that they could be visually verified at some magnification after testing. If a specimen were strained and the coating cracked while the specimen was still in the elastic region, and if the load was removed from the specimen at that time, the crack would close again and not be visually confirmed. This implies a state of compressive residual stress in the coating. It has been speculated that this condition existed in some of the platinum-aluminide coatings where coating spallation of the outer surface layer has been reported [Ref. 37]. Indications are that higher compressive stresses are related to higher surface aluminum and platinum contents and possibly the presence of $PtAl_2$.

For instance, coating No. 3 specimen No. 2, which was tested at room temperature, displayed cracks on the scope at Ø.28% strain, and it was pulled to Ø.3Ø% strain before it was unloaded and examined, but no cracks could be seen at 425x magnification. It was strained again to Ø.58% with similar results, but cracks could still not be seen after

the specimen was unloaded and examined. During a third test conducted on this same specimen it was strained to Ø.77%, and clearly the specimen had permanently elongated by approximately Ø.ØØ4 inch (read on the dial gage after unloading). This time the coating cracks were visible at 425x magnification under the optical microscope.

B. CRACK MORPHOLOGY

The visual results of the coating cracks were exactly as expected and showed beyond a doubt that the cracks had originated in the coatings. All of the photographs in Figure 14 are of coating No. 1 specimen No. 2 which was tested at 81Ø^{o}C (see Table III), and they clearly identify this high activity minimally platinum diffused coating as brittle. The cracks are straight, sharp, and closely spaced, whether viewed from the top or in cross section. Even though the test temperature was high and the substrate was ductile (strained to 3.8Ø% without fracture), the coating cracked early in the test (Ø.36% strain) in a very brittle manner. The cracks are open and very clear in the photographs because of the plastic deformation imparted to the specimen. Notice also that some of the cracks have reinitiated and propagated into the substrate while others are still in the coating only--a clear indication that the cracking was initiated in the coating and not in the substrate.

. Figure 15 displays the ductile nature of the cracks generated in coating No. 5 specimen No. 1. Notice the clear difference between this, the ductile coating, and the brittle coating in Figure 14. At a test temperature of 670°C (see Table III) the cracking is bifurcated and less closely spaced than in the brittle case. This ductile coating required a much higher strain (0.60%) to generate cracks, and even from the side view, the cracks can be seen to bifurcate--characteristic of a ductile type failure mode.

C. THE EFFECT OF PLATINUM ON COATING DBTT

Using the curves generated in Figures 12 and 13 the ductile to brittle transition temperatures for the five coatings tested are presented in Table IV. The data is self consistent and it allows some tentative observations to be made concerning the influence of platinum on diffusion aluminide coatings applied to nickel-base superalloy substrates in regard to the DBTT. Both the inward and outward type diffusion aluminide coatings were embrittled to varying degrees with the addition of platinum, so the claim that platinum-aluminides are relatively brittle is well founded. However, all the outward (HTLA) nickel coatings with platinum were more ductile than the inward (LTHA) aluminum coating even without platinum which is somewhat surprising, since the platinum containing coatings all

exhibit a significant amount of the $PtAl_2$ phase which is regarded as brittle. This indicates that aluminum content may be more critical than previously believed. The data also attests to the importance of the processes used to form the coatings in determining their mechanical properties, but since both aluminum and platinum levels change with heat treatment and processing, it is difficult at this point to sort out the specific effects of one or the other. More detailed analysis of the coating compositions is required.

Note that all the outward nickel coatings with platinum had DBTT's well below the estimated range for $PtAl_2$ projected with Lowrie's formula (870°C - 1070°C), while the inward aluminum coatings with platinum may very well have DBTT's in the projected range, as this testing only ascertained that they were greater than 810°C. The point is stressed that Lowrie's formula should be used only as a guideline when discussing coating ductility. Actual coating-substrate systems must be tested to ascertain their specific mechanical properties because of the metastable nature of the systems and the strong influence of heat treatments and aluminizing processes on the structure and properties of the platinum-aluminides. Indications are clear that an increased aluminum content as a part of $PtAl_2$ (coating Nos. 1 and 3) can cause a brittle structure while

the $PtAl_2$ as a part of a second phase in a presumably more ductile matrix does not tend to reduce coating ductility (coating No. 5). The presence of platinum in solution in the β(NiAl) coating matrix appears to reduce ductility somewhat in all cases (coating Nos. 1-5).

While additional testing is warranted to confirm these results, it is felt that a viable and economical method for determining the ductile to brittle transition temperature of turbine blade coatings is presented here. Even though temperatures could not be reached to ascertain the DBTT's of the high activity inward aluminum coatings with platinum, it was confirmed that they were above that of the conventional inward aluminum coating without platinum.

IV. CONCLUSIONS AND RECOMMENDATIONS

Based on the results of tensile tests conducted on the five platinum-aluminide coating types on IN738, the following conclusions can be drawn:

1) A valid method for determining the DBTT of coating-substrate systems has been established using U.S. Naval Postgraduate School equipment.

2) Because the pre-aluminizing heat treatment and the aluminizing treatment greatly effect the structure and composition of platinum-aluminide coatings, each of the structures display characteristically individual ductility properties.

3) Although further tests should be conducted, the low activity high platinum diffusion coating on IN738 is the most ductile platinum-aluminide coating tested in this study.

4) A significant level of residual compressive stress was observed in all platinum-aluminide coatings studied.

Recommendations for further study are:

1) A furnace which will allow tensile testing up to 1100°C should be obtained so that the DBTT's of the high activity inward coatings with platinum on IN738 may be ascertained as well as the high end of the other DBTT curves.

2) More tests should be conducted using the substrate and coatings employed in this thesis so that some data scatter can be obtained and used to further define the conclusions of this study.

3) This testing method should be used to test more substrate-coating combinations to further define the properties of the platinum-aluminides.

4) Microprobe analysis of the coating structures should be used in further testing to determine the exact content and effect of platinum and aluminum in each phase of the coatings.

5) The implication of coating residual stress in all the platinum-aluminides tested warrants further study in the context of its effect on coating processing, handling, and thermal fatigue testing.

APPENDIX A: TABLES I-IV

TABLE I

IN738 Composition (Weight Percent)

Ni	Cr	Co	Mo	W	Ti	Al	Nb	Ta	C
60.42	16.0	8.5	1.75	2.6	3.4	3.4	0.9	1.75	0.17

B	Zr	Fe	Mn	Si
0.01	0.10	0.5 max	0.2 max	0.3 max

TABLE II

Platinum-Aluminide Coatings Formed on IN738 Substrates

Coating	Platinum Diffusion	Aluminizing and Post Heat Treatment
No. 1	870°C / 1/2 hour	LTHA*
No. 2	980°C / 2 hours	HTLA + 1080°C/4 hrs
No. 3	1052°C / 1 hr	LTHA*
No. 4	1052°C / 1 hr	HTLA + 1080°C/4 hrs
No. 5	1080°C / 4 hrs	HTLA + 1080°C/4 hrs

* LTHA conducted in most industrial applications includes a post heat treatment of 1080°C/4 hrs..

TABLE III

Coating Strain to Failure Data*

Coating	Specimen	Test Temperature (°C)	Strain to Coating Crack (%)	Total Strain Imparted to Specimen (%)	Specimen Fractured
1	1	21	0.36	0.60	Yes
	2	810	0.36	3.80	No
2	1	860	---	1.71	No
	1	21	0.50	0.70	No
	2	660	0.50	2.00	No
	3	750	0.61	1.20	No
3	1	810	0.25	2.20	Yes
	2	21	0.28	0.77	No
	3	300	0.28	0.71	No
	4	550	0.25	0.64	Yes
4	1	800	0.66	1.10	No
	2	21	0.45	0.75	Yes
	3	610	0.45	0.65	Yes
	4	660	0.45	0.65	Yes
5	1	670	0.60	1.10	No
	1	21	0.25	0.65	No
	2	560	0.40	0.70	No

*Does not include initial embrittled failures which did not yield data.

TABLE IV

The Ductile to Brittle Transition Temperatures (DBTT)*

Coating	Type	DBTT
1	high activity, inward Al, low Pt diffusion	>810°C
2	low activity, outward Ni, medium Pt diffusion	720°C
3	high activity, inward Al, medium Pt diffusion	>810°C
4	low activity, outward Ni, medium Pt diffusion	750°C
5	low activity, outward Ni, high Pt diffusion	640°C
	high activity, inward Al, no Pt	840°C**
	low activity, outward Ni, no Pt	580°C**

* temperature at which 0.6% strain causes coating compromise

**Goward data [Ref. 36]

APPENDIX B: FIGURES 1-15

Figure 1. Inward Nickel-Aluminide Coating (1ØØØx)

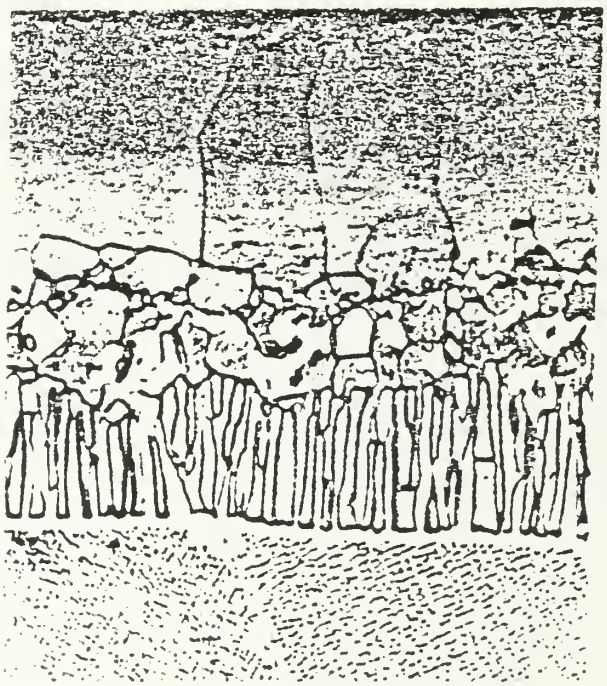

Figure 2. Outward Nickel-Aluminide Coating (1ØØØx)

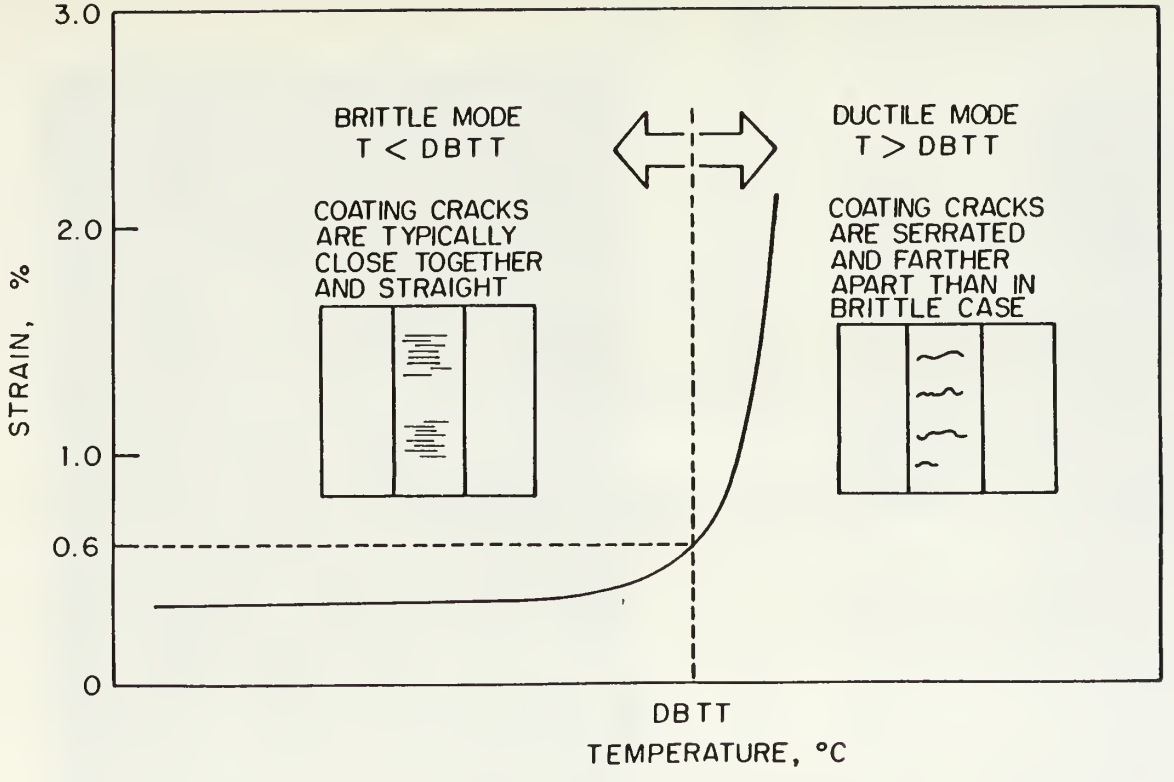

Figure 3. Typical Ductile to Brittle Transition Temperature Graph for a Given Coating-Substrate System

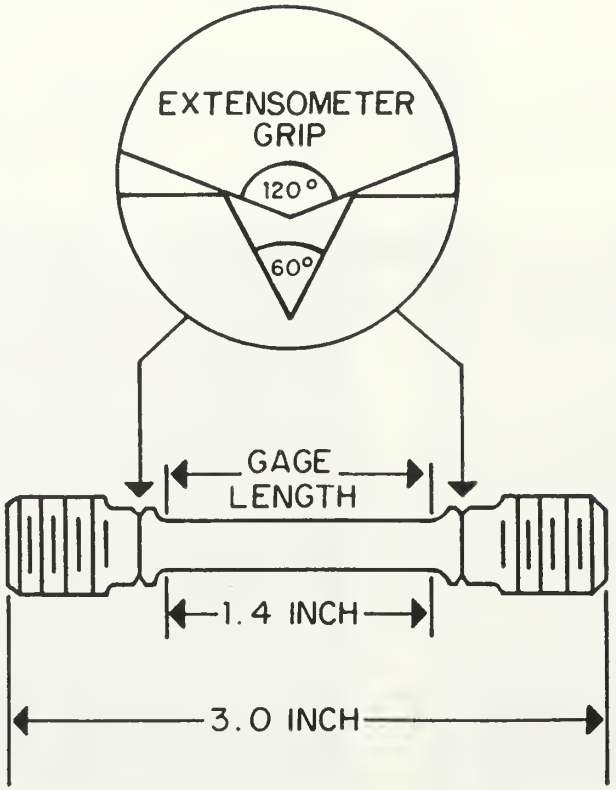

Figure 4. Coated Tensile Specimen

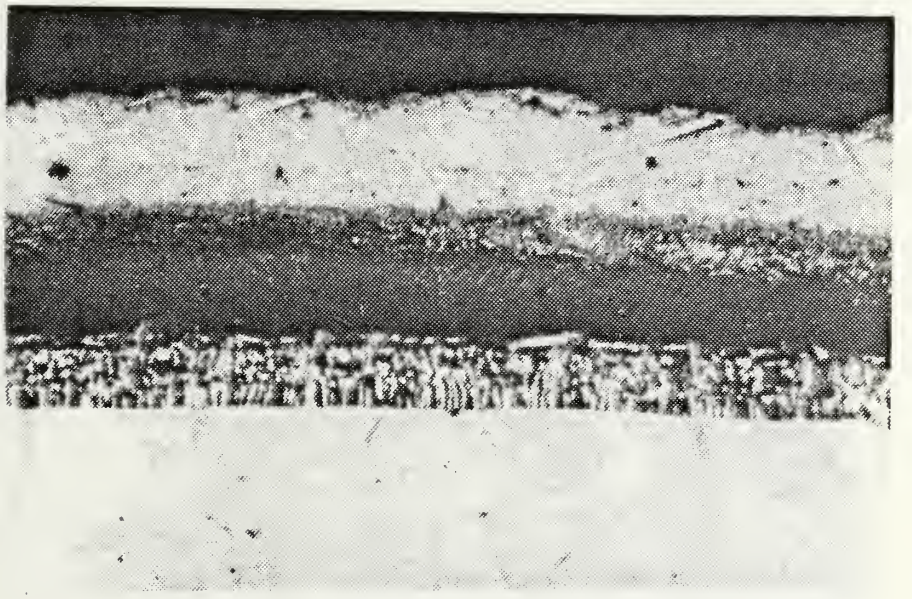

Figure 5. Coating No. 1 - Low Pt Diffusion, Inward Al, Etched (425x)

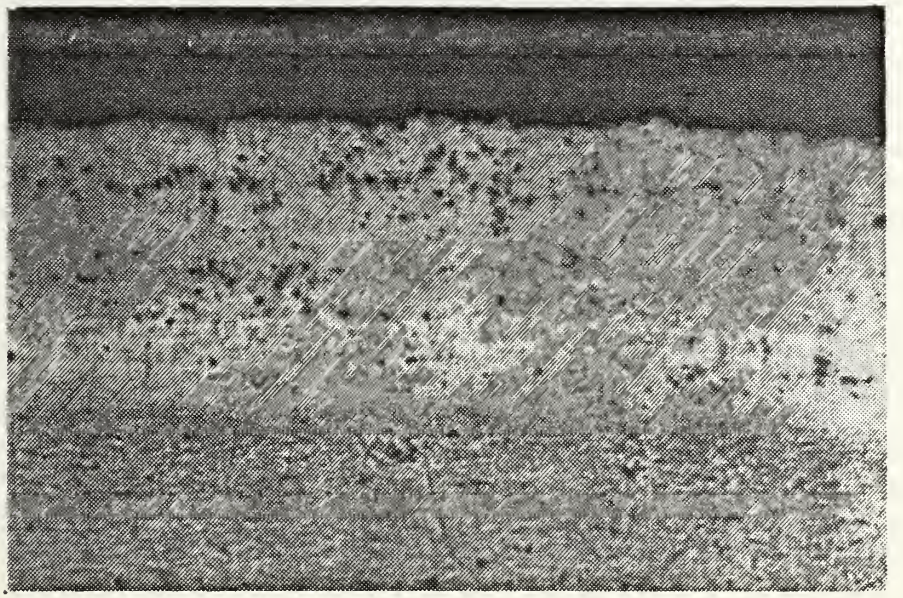

Figure 6. Coating No. 2 - Medium Pt Diffusion, Outward Ni, Etched (425x)

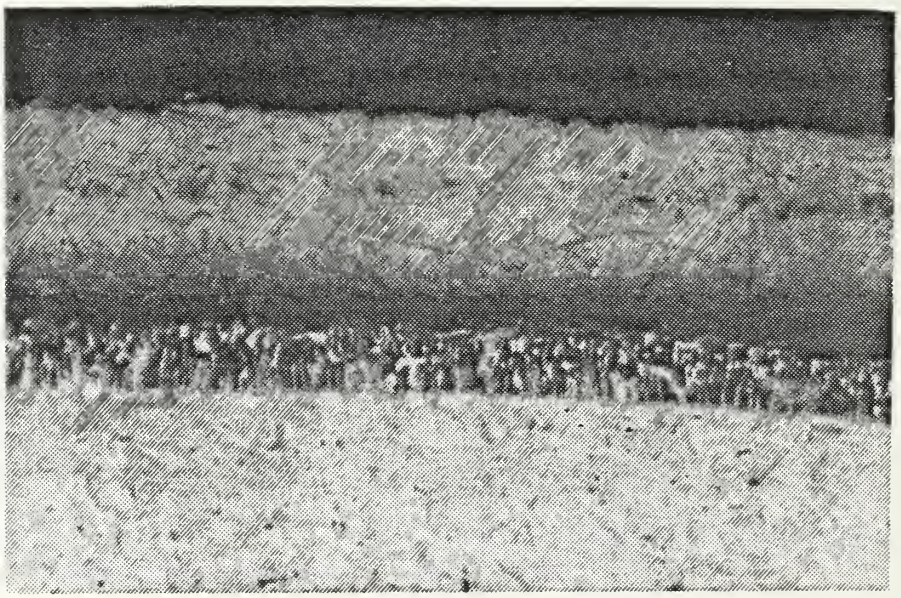

Figure 7. Coating No. 3 - Medium Pt Diffusion, Inward Al, Etched (425x)

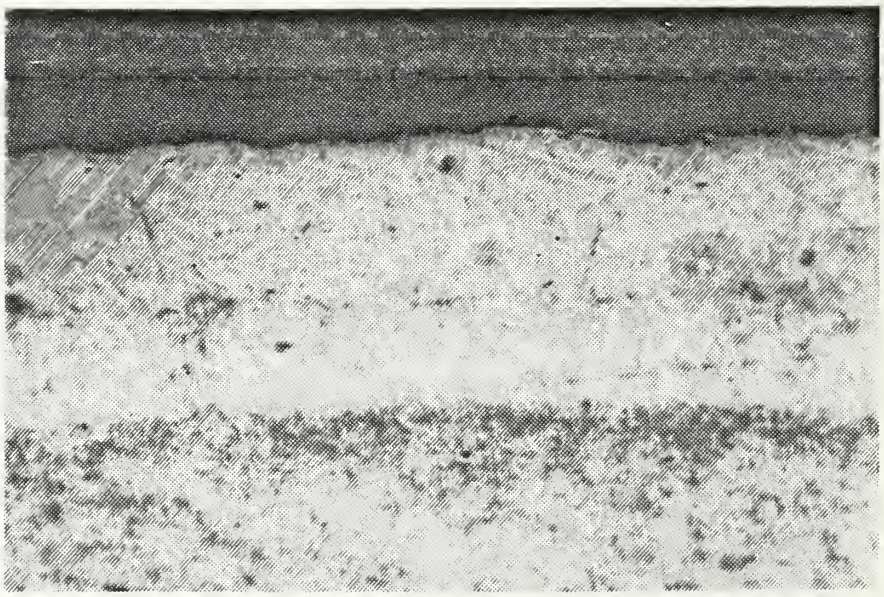

Figure 8. Coating No. 4 - Medium Pt Diffusion, Outward Ni, Etched (425x)

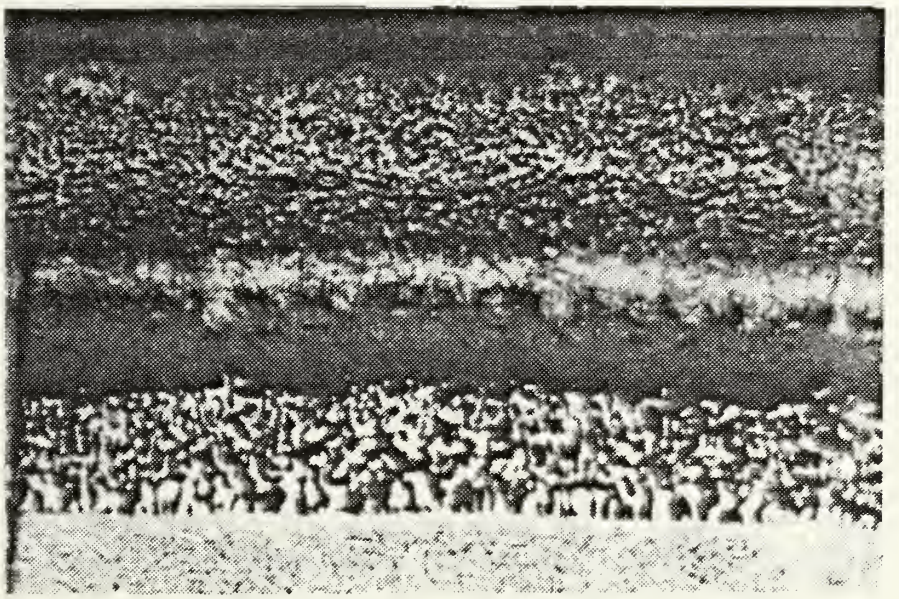

Figure 9. Coating No. 5 - High Pt Diffusion, Outward Ni, Etched (425x)

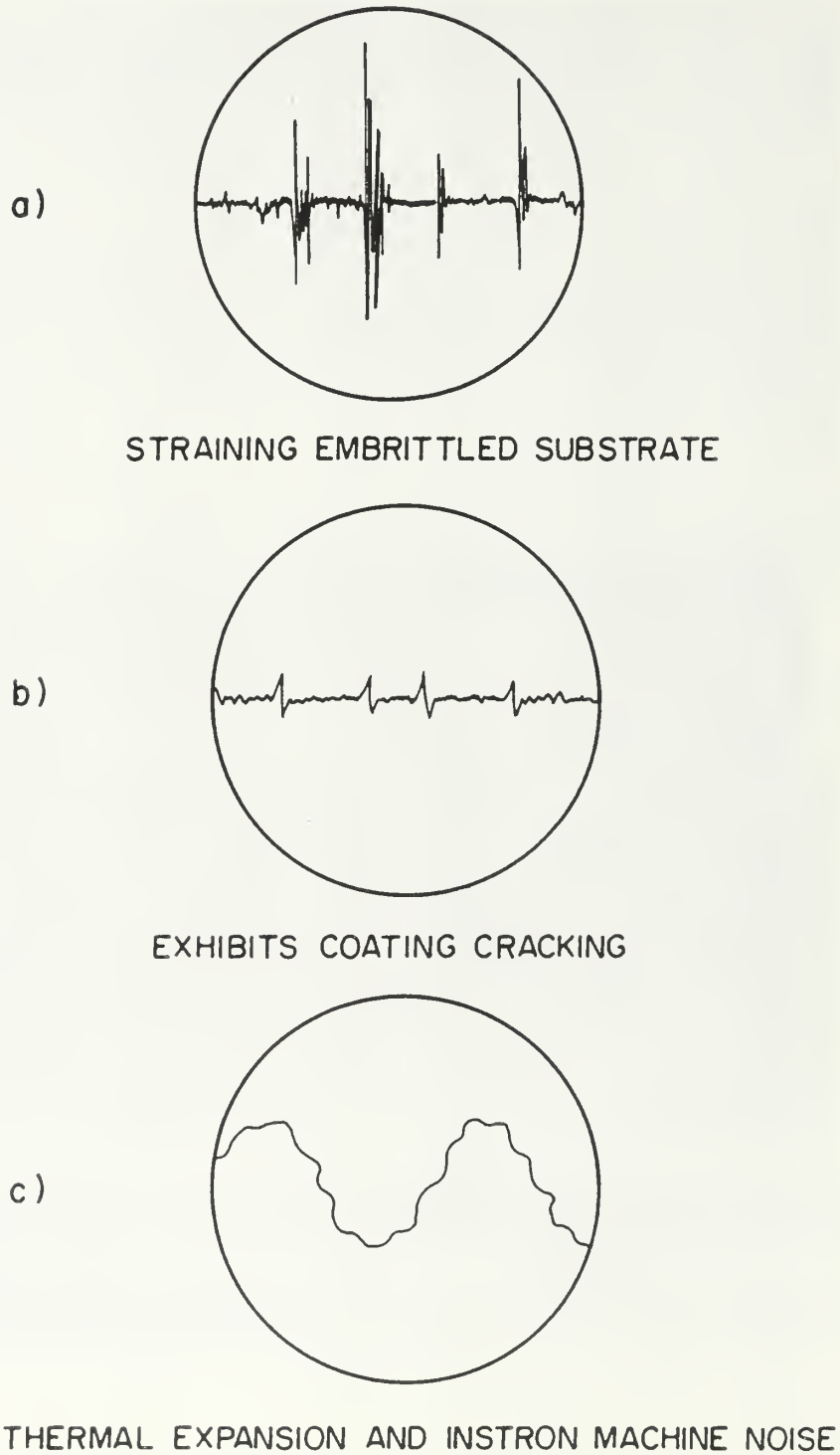

Figure 10. Typical Oscilloscope Signatures During Tensile Testing

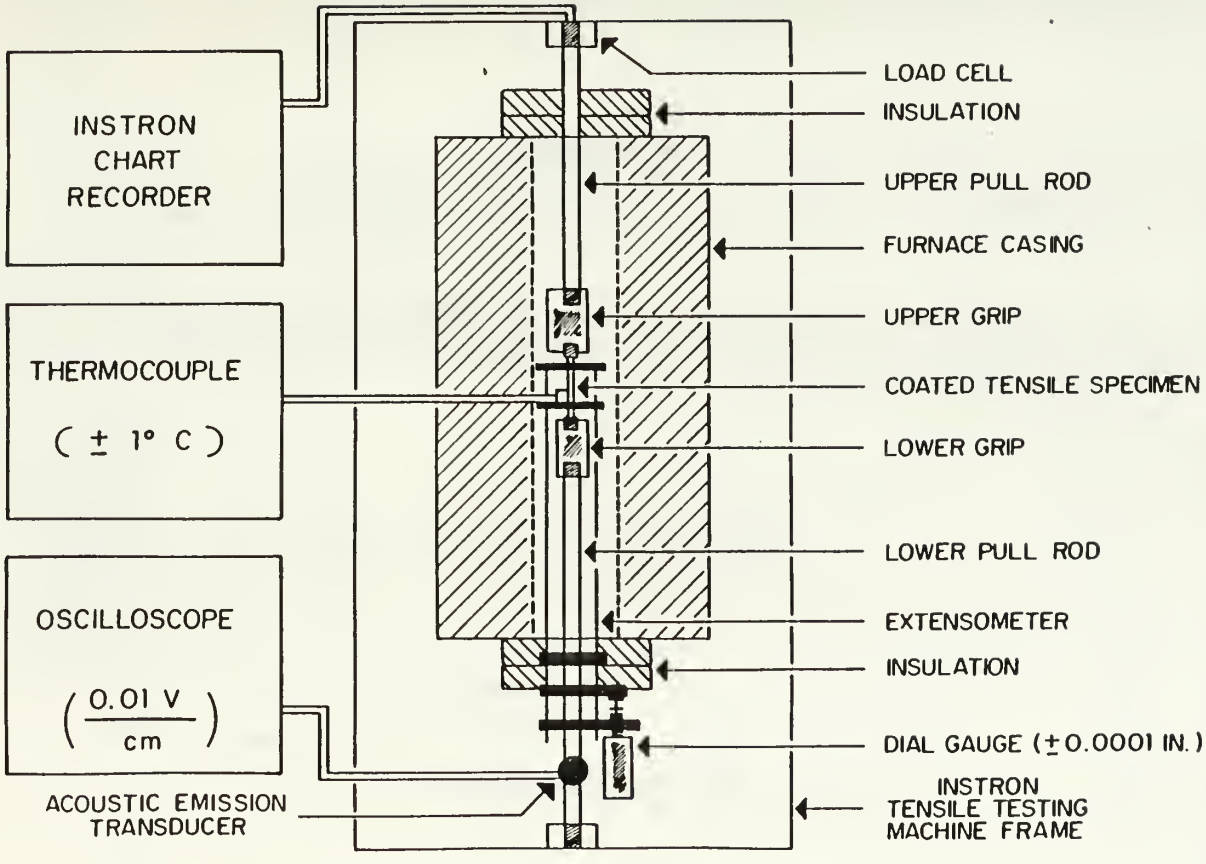

Figure 11. Experimental Set-Up for Coating-Substrate Ductility Testing

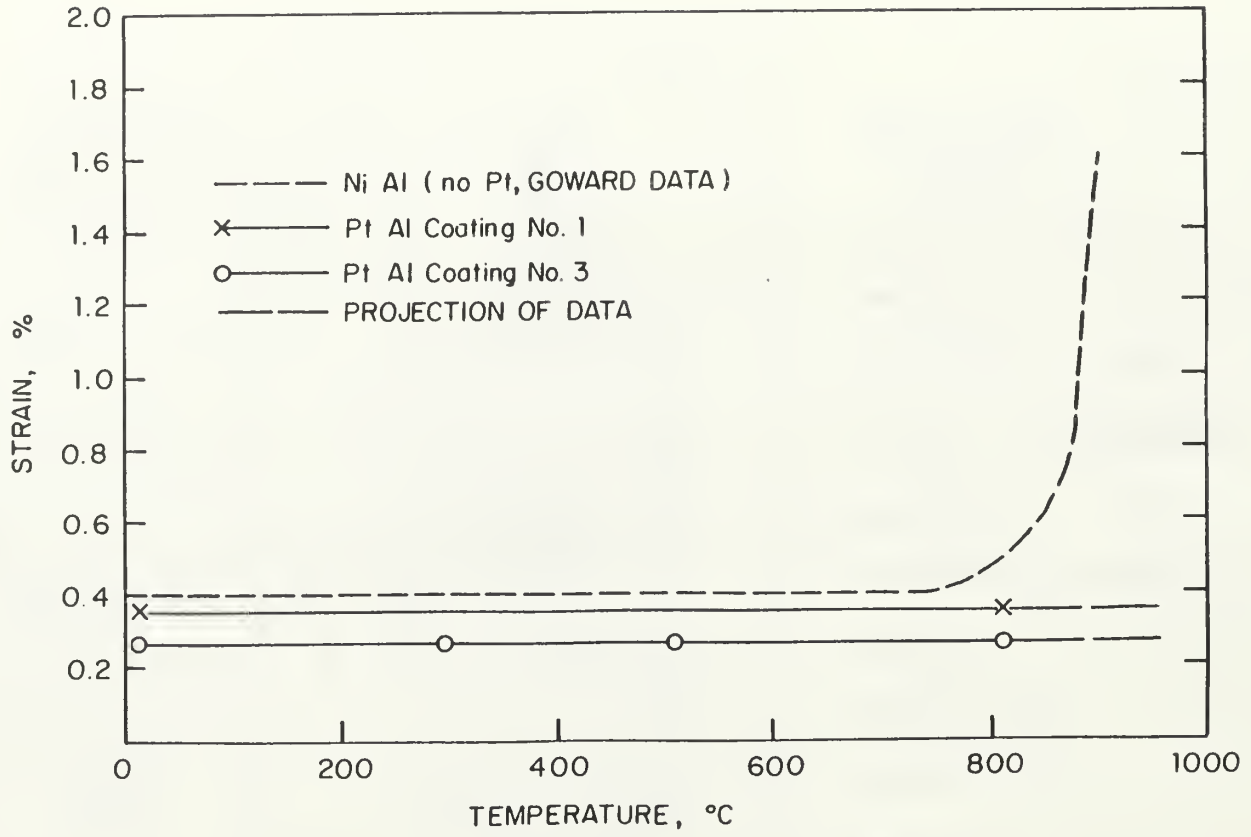

Figure 12. Ductility Graph of Inward Aluminum, High Activity Coatings (LTHA)

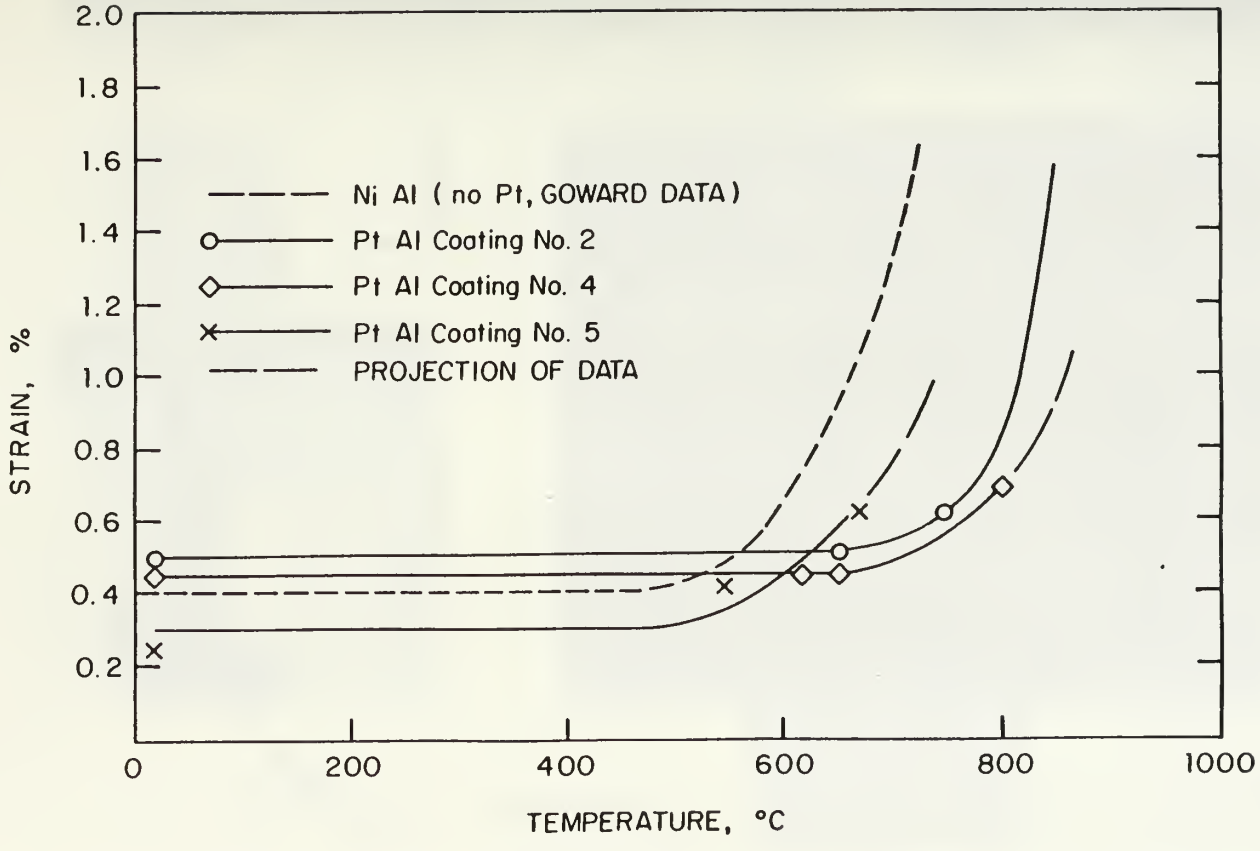

Figure 13. Ductility Graph of Outward Nickel, Low Activity Coatings (HTLA)

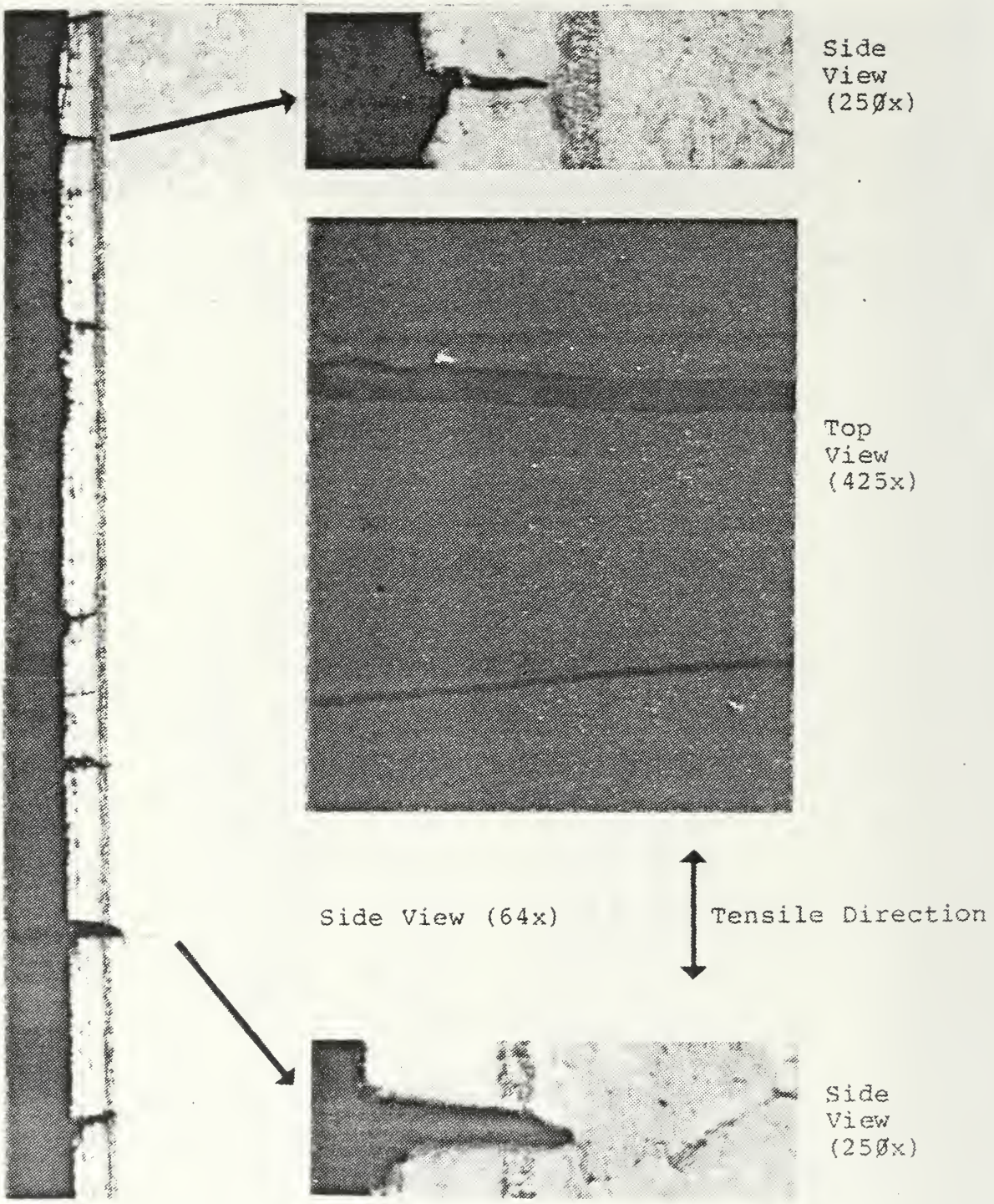

Figure 14. Brittle Failure of Coating No. 1

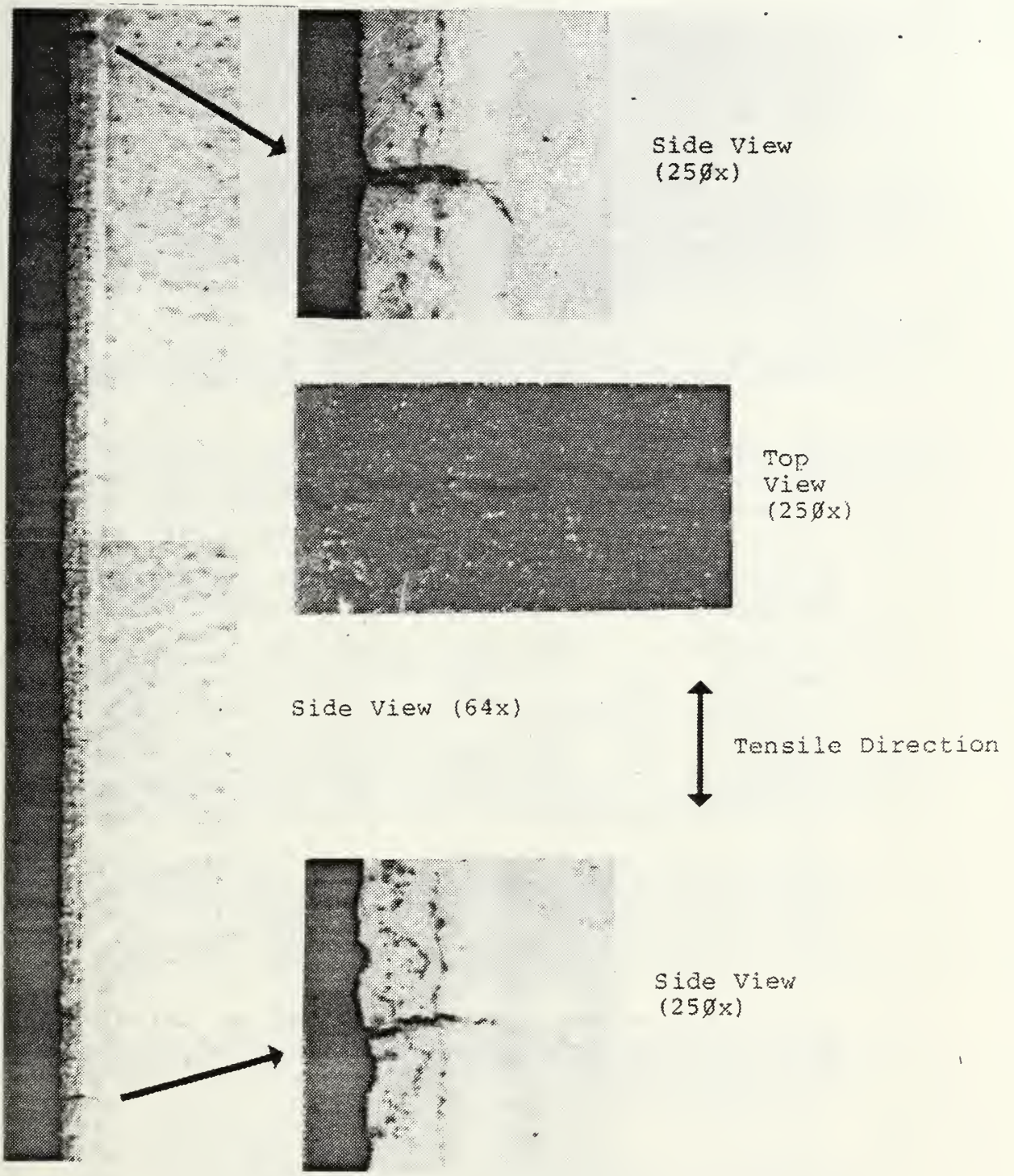

Figure 15. Ductile Failure of Coating No. 5

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