NDE of PWA 1480 Single Crystal Turbine Blade Material

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ABSTRACT

Cantilever bending fatigue specimens were examined by fluorescent liquid penetrant and radioactive gas penetrant (Krypton) NDE methods and tested. Specimens with cast, ground, or polished surfaces were evaluated to study the effect of surface condition on NDE and fatigue crack initiation. Fractographic and metallurgical analyses were performed to determine the nature of crack precursors. Preliminary results show that fatigue strength was lower for specimens with cast surfaces than for specimens with machined surfaces. The liquid penetrant and gas penetrant techniques both provided indications of a large population of defects on the cast surfaces. On ground or polished specimen surfaces, the gas penetrant appeared to estimate the actual number of voids more accurately than the liquid penetrant.

INTRODUCTION

The need for increasing the life and reliability of turbine blades in SSME turbopumps has led to a search for an alternative material for this application. The single crystal material, PWA 1480, is a leading candidate for blades in advanced turbopump designs because of its improved resistance to hydrogen embrittlement and increased notched bar tensile strength, relative to the directionally solidified MAR-M246+Hf superalloy currently being used (ref. 1). While carbides have been the primary crack precursor in MAR-M246, fractographic observations have shown that fatigue cracks in PWA 1480 are frequently initiated by irregularly shaped pores generated during casting (crystal growth). Although porosity can be greatly reduced by hot isostatic pressing, the potential still exists for voids with chord lengths over 50 µm to contribute to early failure. Until processing technology is developed to totally eliminate these defects, the cast components in which they reside must be identified and a decision to reject them must be based on nondestructive examination. This presents a challenge to NDE technology which heretofore has not been commonly used to detect defects much smaller than 250 microns.

Radiography, used primarily to detect internal flaws, is insensitive to voids with dimensions less than one percent of the thickness of the component being evaluated. Fluorescent liquid penetrant techniques are the most common methods used to inspect for surface defects in turbine blades. Liquid penetrants require that the defect be surface connected and large enough to permit the liquid to partially fill the volume. A large void barely open to the surface will not be detected if the liquid cannot enter. Thus, methods with greater sensitivity must be investigated to complement liquid penetrants and X-rays. The sensitivity requirement also places emphasis on the surface condition of the component because of the possibility of camouflaging small defects by texturing or by a machining practice that produces a thin layer of plastically deformed metal. This paper presents preliminary results of a program to evaluate a relatively new technique with greater sensitivity potential and addresses the effect of surface condition on NDE as well as fatigue strength. The study examines a little used radioactive gas penetrant method referred to as the krypton evaluation technique (KET). A comparison is made with a fluorescent penetrant technique used on aircraft engine turbine blades and SSME turbopump blades. Liquid and gas penetrant examination was performed on fatigue specimens to determine the relationship between NDE indications and subsequent fatigue crack initiation sites.

MATERIAL AND TEST SPECIMENS

MATERIAL - Single crystal PWA 1480 material was procured from Howmet Corporation in the form of slabs, nominally 7.6 by 5 by 0.64 cm thick, cast from a single heat using a standard thermal gradient established by the
vendor. The chemical analysis was: 10.0Cr, 4.8Al, 1.4Ti, 11.8Ta, 4.2W, 5.3Co, balance Ni, in weight percent. The specified crystallographic orientation required the long axis of the slab to be within 10° of the <100> direction. Heat treatment after casting consisted of a solution treatment at 1290 °C, precipitation treatment at 1080 °C, and aging at 870 °C. Part of the batch was later hot isostatically pressed (HIPed) at 1290 °C at pressures ranging from 35 MPa to 103 MPa. After HIPing, the slabs were again heat treated following the same parameters used after casting.

Essentially no carbides or borides are present in single crystal materials because C, B, and Zr are not added for grain boundary strengthening (ref. 2). Interdendritic porosity, however, is an inherent part of the PWA 1480 microstructure in the as-cast condition. The average pore diameter is less than 10 µm, but there are a substantial number of voids with high aspect ratios and dimensions an order of magnitude greater than the average size (fig.1). These relatively large voids are of concern because they have been found to initiate cracks in test specimens (ref.3). HIPing can reduce the average size and population of internal pores. Metallographic crossections from five randomly chosen HIPed fatigue specimens from this program revealed little evidence of internal voids at magnifications up to 200X. An example of a typical unetched crossection of a HIPed sample in figure 1 shows no voids. It should be noted that HIPing cannot close pores open to the surface so the possibility still remains for many voids to exist in parts of blades with little or no surface material removed.

SPECIMENS AND TESTING - Cantilever plane-bending fatigue specimens were machined from both as-cast and HIPed slabs to specifications shown in figure 2. The design provides for a constant stress test section over an area of 1.25 cm². To study the effect of surface condition on fatigue life and the ability of NDE to detect the voids that contribute to crack initiation, specimens with three surface conditions were prepared. These included HIPed and unHIPed specimens with an unfinished surface (essentially as-cast except for subsequent thermomechanical treatments), machined specimens finished to nominally 0.2 µm RMS by grinding, and polished specimens prepared by grinding to a similar finish and lapping with alumina polishing compound to remove some of the cold

Figure 1.—Typical pore distribution in HIPed and unHIPed PWA 1480 single crystal material. No pores were observed in HIPed material at these magnifications. Unetched crossections normal to the <100> direction.
work induced by the previous step (grinding marks were not entirely removed).

Fatigue testing was performed at room temperature in a computer controlled, hydraulically actuated machine. The large end of the specimen was clamped in a vise positioned such that the small end was in line with the axis of the loading ram. Tests were load controlled in the elastic range of the material. Cyclic tensile bending loads were applied to the surface of interest at a minimum-to-maximum stress ratio of 0.05. The intent was to initiate cracking in a reasonable time with no initial plastic deformation and to preserve the fracture surfaces for fractographic analysis. A comprehensive fatigue study was not an objective of the program. The travel distance of the ram was monitored and limits were set to stop cyclic loading as soon as the specimen deflection increased by 5 percent from the value at the start of the test. At this time a small crack was usually visible with the unaided eye on the tensile surface of the sample. The presence of such a crack defined specimen failure. The crack location was noted, the deflection limit removed, the mean load increased, and testing continued until complete fracture occurred (on the upstroke of the ram to prevent the fracture surfaces from making contact).

NONDESTRUCTIVE EVALUATION

Two nondestructive evaluation methods will be discussed in this paper; fluorescent liquid penetrant and radioactive gas penetrant. These methods are theoretically sensitive to surface connected flaws only, since they require a certain amount of penetrating fluid to enter the discontinuity. The procedures for carrying out the examination of an object and the methods of observation and recording the results differ substantially. A brief description of the techniques follows, with detailed technical information given in references 4 and 5.

LIQUID PENETRANT - Successful liquid penetrating inspection depends on the ability of a liquid to wet the surface of a solid object with a continuous film that is drawn into surface discontinuities such as cracks and pores by capillary action. Figure 3 schematically illustrates the process. The object must be cleaned to optimize wetability and to eliminate absorbent foreign matter that would create false indications. Vapor solvent degreasing is very effective for metallic objects. The part is then immersed in the penetrating liquid, and after a suitable dwell time the excess is washed from the surfaces, leaving residual penetrant only inside the discontinuities. A developer, consisting of a fine absorbent powder, is then applied to wick the liquid from the discontinuity to the surface in the immediate vicinity of the defect. The most sensitive penetrants contain particles that fluoresce in the presence of ultraviolet light, which highlights the flaw in darkened surroundings. A wide variety of penetrating liquids, dyes, fluorescent materials, and developers are available, and it is important to choose the optimum combination for a given application.

Examination of specimens for this program was performed at the Department of the Air Force, Wright Laboratory. A post-emulsifiable hydrophilic penetrant (very bright, level 4) was used with a dry powder developer. Penetrant dwell time was ten minutes, followed by prewashing with water until virtually all of the penetrant was removed from the surfaces. Specimens were then dipped into an emulsifier (1-2 seconds for machined and polished surfaces, and five minutes for cast surfaces) and rinsed with water to remove final traces of penetrant from the surfaces. Specimens were then dried five minutes at 65 °C and developed for a minimum of 10 minutes. Indications were photographically recorded under ultraviolet light.

RADIOACTIVE GAS PENETRANT - The penetrating liquid and radioactive gas techniques are similar in principle, but the procedures differ because of the greater mobility and radioactivity of krypton gas. The radioactive gas penetrant technique is illustrated in figure 4. The test object is cleaned and placed in a controlled environment chamber, and a vacuum pumped to remove adsorbed air molecules from all surfaces, particularly in the defect. The chamber is backfilled with dilute (less than 5 percent) krypton-85 gas which is adsorbed onto the object and defect surfaces. The krypton is then pumped from the chamber and the test object removed. The adsorbed gas on the object is quickly released into the atmosphere of the
chamber when it is pumped down, normally to a pressure of 0.1 mm Hg. Krypton gas remains entrapped in tight defects long enough to apply beta radiation sensitive film or emulsion to the surface and produce an autoradiograph showing the exact location and, to lesser degree, the relative volume of the defect. Shallow flaws with smooth sides can be missed if the surface opening is so great that the krypton molecules escape before film is applied. This limitation, coupled with the small diameter of the krypton molecule (0.3 nm) makes the method best suited to detection of relatively tight flaws. Thus, it may be most effectively used to complement rather than replace liquid penetrants. Gas penetrant evaluation of the fatigue specimens in this investigation was performed by Qual-X, Incorporated, the sole provider of this service in the United States.

RESULTS AND DISCUSSION

The following paragraphs describe results of destructive and nondestructive tests of PWA 1480 fatigue specimens evaluated in this investigation. The data presented show results of liquid and gas penetrant examination of fatigue specimens with various surface preparations prior to fatigue testing. Fractographic and metallographic observations are shown only for samples with cast surfaces; results for specimens with machined and polished surfaces were not available at the time of this writing. Plane bending fatigue test data for cast and machined specimens are also presented.

LIQUID PENETRANT INSPECTION - Results of fluorescent liquid penetrant inspection of specimens with cast and machined surfaces are shown in figures 5 and 6 respectively. A high level of fluorescence is present in most cast specimens, typical of rough surfaces and a large surface pore population. The sample-to-sample variation indicates that subtle processing variations took place during casting or subsequent handling. Each specimen has a character of its own and no systematic differences appear to exist between the HIPed and unHIPed material. The machined specimens in figure 6 have a much lower background noise level and exhibit a relatively small number of discrete indications in clear contrast to the cast surfaces. The ground specimens also exhibit no systematic differences between HIPed and unHIPed material. Comparisons with metallographic evidence in figure 1 suggest that there are fewer indications than might be expected in the unHIPed samples, probably because the liquid cannot penetrate into voids unless they are significantly larger than 10 µm. On the other hand, the HIPed sample results show more
indications than would be expected from the metallographic evidence (fig. 1). More evidence is required to determine whether the latter are true or false indications. Liquid penetrant results on polished specimens did not differ substantially from the ground specimen results and therefore examples are not shown here.

**GAS PENETRANT INSPECTION - Autoradiographs**

of specimens with cast, ground, and polished surfaces are presented in figures 7 to 9 respectively. The bright spots and areas are where the krypton gas was retained, resulting in darkening of the film placed in contact with the specimen surface. The prints in the figures show the inverse contrast to the original film. Figures 7 and 8 show results for the same specimens evaluated by liquid penetrant in figures 5 and 6. It is obvious that the cast specimens have far greater affinity for krypton gas than either the machined or polished surfaces. It is impossible to identify discrete indications on the cast surfaces. Five of the specimens (fig. 7) have white stripes running in random directions across the large end, which coincide with identification marks made by an indelible marking pen. Even though evidence of the marks was removed before NDE, the regions seem to have an affinity for krypton molecules. The cast surfaces in general retained krypton gas for a longer period of time than machined or polished surfaces, and had to be heated to drive off the adsorbed gas and get the level of radiation down to near background levels.

The autoradiographs of machined samples in figure 8 are very different from those of cast samples in that the indications are discrete and relatively few in number. This is undoubtedly because a layer of relatively porous surface material was removed by grinding. Because of their small size some of the indications may not have reproduced well for publication, therefore, the observations will be described in text. There is a striking difference in the

![Figure 7](image7.png)  
**Figure 7.—Radioactive gas penetrant autoradiographs of specimens with cast surfaces. High level of gas retention by surface porosity and rough texture causes general exposure of film, making detection of discrete defects difficult.**

![Figure 8](image8.png)  
**Figure 8.—Radioactive gas penetrant autoradiographs of specimens with machined (ground) surfaces. HiPed specimens exhibit very few indications, implying that internal porosity is virtually eliminated by hot isostatic pressing.**

![Figure 9](image9.png)  
**Figure 9.—Radioactive gas penetrant autoradiographs of specimens with polished surfaces. Comparison of un-HiPed samples with those in figure 8 suggests that polishing enhances detection of smallest pores by removing cold worked surface.**
number of indications in the unHIPed material compared to the HIPed material. The two HIPed samples on the right have two indications in the test section and a dozen or more in the large end, while the other two have no indications in the test section and very few in other parts of the specimen. The unHIPed specimens have countless indications throughout, which agrees generally with the metallographic observations described in an earlier section, insofar as the relative population of voids in the two materials is concerned. It should be noted that, although the specimens were identical, virtually none of the indications produced by the gas and liquid penetrants are in the same location on the surface. At this point there is insufficient analysis to determine whether the methods complement each other or if one or both are producing false indications.

Figure 9 shows autoradiographs of HIPed and unHIPed specimens that were polished to remove the thin layer of disturbed (smeared) material usually caused by grinding. The results for HIPed material are similar to the ground surface results in figure 8, again confirming metallographic evidence that little porosity exists. However, the data for unHIPed material show somewhat more indications on polished samples than on as-ground samples. Because the specimens are different, it is possible that the polished set simply contains more voids, but there certainly is an implication that polishing enhances void detection by the gas penetrant. A similar effect was not evident on the same specimens with liquid penetrants, which produced few indications on both polished and ground surfaces. Thus, in general, the radioactive penetrant technique appears to provide more accurate information regarding the existence and distribution of very small pores.

METALLOGRAPHY AND FRACTOGRAPHY - An example of the type of crack origin observed in specimens with cast surfaces is shown in figure 10. Because of a preponderance of defects at the cast surfaces, cracking always initiated at a point where a concentration of defects occurred. Figure 10 shows that the fatigue crack leading to fracture started in the vicinity of three defects, one of which may be an inclusion while other two have a different character. Instead of voids, they resemble pockets of extremely fine porosity. Metallographic crosssections were taken from the ends of ten specimens for further study. Typical results are presented in figure 11 which shows closely spaced reaction zones along the cast surface that at low magnifications appear to be incipient melting, which is always of concern when solution treating or HIPing. At higher magnifications, however, pockets containing a fine granular structure beneath a skin-like layer are revealed. The structure has the appearance of γ' with the γ phase leached out. Energy dispersion scans (EDS) showed that the particles indeed are the γ' phase. The size and number of these pockets was similar for HIPed and unHIPed samples. Similar features were not observed internally or at a machined surface.

Figure 11 also shows a subtle difference in the thickness of the skin on the HIPed and unHIPed materials. The HIPed samples had a dual layer skin that was a total of about 5 µm thick. EDS shows that the composition of the inner layer is identical to the γ' particles. The outer skin layer is similar to the γ phase except that it is higher in tantalum and, unlike γ, has a plate-like structure. EDS showed alternating plates to be essentially Ni₅Ta. The unHIPed samples had a much thinner skin, 1 µm or less, but a reliable analysis was not obtained. An example in figure 11 illustrates that these pockets may all be exposed to the surface through breaks in the skin. It was surmised that phosphoric acid grain etchant used to confirm that the casting is a single crystal, seeped under the skin and formed the reaction zones by leaching out the matrix because it could not be washed out.
Reaction zone distribution

Figure 11.—Reaction zones near cast surfaces containing precipitate particles of γ' phase. γ matrix appears to have been leached out by a grain boundary etchant. Occurrence of reaction zones is similar in HIPed and unHIPed material.

Figure 12 shows etched cross-sectional views at the cast surfaces of HIPed fatigue specimens. The primary features are areas of recrystallized material along the cast edge, which are discontinuous and vary in depth up to 75 μm. The high magnification photographs confirm that the interface between the light and dark regions are grain boundaries. Since the alloy does not contain grain boundary strengtheners, it is expected that material containing these unwanted features would be relatively weak, especially where the grain boundary is oriented normal to loading direction. Recrystallization was not observed in the unHIPed specimens examined.

FATIGUE TESTS - A comparison of the fatigue life of PWA 1480 specimens with cast and machined surfaces is presented in figure 13. Both HIPed and unHIPed materials are included without distinction. It is clear that the machined samples have a higher fatigue strength than specimen with cast surfaces. This is probably due to the removal of the large population of γ matrix-free reaction zones that exist near as-cast surfaces as described previously. Although the dimensions of individual zones are small, collectively they probably constitute a significant stress concentration that leads to early cracking.

Figure 14 compares the fatigue strength of specimens in the HIPed and unHIPed conditions, with cast surfaces only. Taken as a whole, the two sets of data suggest a subtle difference in fatigue strength for the two material conditions and imply that HIPing has a weakening effect. Since both HIPed and unHIPed specimens contain γ free reaction zones, their existence alone cannot explain a difference in strength. As noted in the previous section the HIPed material was recrystallized along the cast surface. It is very likely that the combined negative effect of the reaction zones and the recrystallized areas results in slightly lower average fatigue strength for the HIPed specimens. The limited observations of this investigation suggest that a minimum of 75 μm must be carefully removed from cast surfaces to remove the reaction zones due to etching, and the recrystallized areas formed during HIPing, in order to achieve mechanical properties representative of the bulk material.
Recrystallization along cast surface

Discontinuous recrystallization

Grain boundary

Cast edge and grain boundary

Figure 12.—Crosssections of HIPed specimens showing recrystallization near the cast surface. Recrystallization was discontinuous and observed only in HIPed material. Depth ranged up to 75 µm.

Figure 13.—Cantilever bending fatigue results on specimens with either machined or cast surfaces. Fatigue strength is improved by removing the cast surfaces.

Figure 14.—Cantilever bending fatigue results on HIPed and unHIPed specimens with cast surfaces. HIPed specimens have slightly lower fatigue strength.
SUMMARY OF RESULTS

1. Void size in the unHIPed PWA 1480 material averaged less than 10 µm but a significant number with dimensions an order of magnitude greater were in evidence. HIPing virtually eliminated internal voids but did not eliminate discontinuities open to the surfaces.

2. The radioactive gas penetrant technique using krypton 85 was more sensitive to small voids than the fluorescent liquid penetrant technique. The gas penetrant results agreed with microstructural observations of PWA 1480 internal pore distribution in that many indications were produced on unHIPed specimens and very few on HIPed samples. The liquid penetrant appeared to underestimate the number of voids in unHIPed samples.

3. The liquid and gas penetrants both detected the high level of porosity in specimens with cast, unfinished surfaces. The resultant background noise made it virtually impossible to observe discrete defects with either method.

4. Polishing of machined surfaces appeared to enhance detectability of voids by the gas penetrant. A similar effect was not observed with liquid penetrant.

5. The cast surfaces of fatigue specimens contained a profusion of small reaction zones containing γ' particles but lacking the γ matrix. It appears that the matrix was leached out by the phosphoric acid etchant used to check for possible grain boundaries. The size of the matrix-free pockets ranged up to about 50 µm.

6. The cantilever bending fatigue strength of specimens with cast surfaces was lower than for specimens with machined surfaces in both the HIPed and unHIPed conditions. Fractographic evidence indicated that the probable cause was the presence of γ free reaction zones at the cast surface.

7. The HIPing cycle used in this investigation produced a narrow discontinuous zone of recrystallization along the cast surface. The fatigue strength of HIPed specimens was slightly lower than for unHIPed specimens with cast surfaces.

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