NONDESTRUCTIVE QUANTIFICATION OF INDUCED SURFACE TREATMENT AND RELAXATION EFFECTS IN METALS

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Abstract: Photon Induced Positron Annihilation (PIPA) and related portable technologies' have demonstrated their ability to nondestructively quantify shot peening/surface treatments and relaxation effects in single crystal superalloys and steels with a single measurement as part of a National Science Foundation SBIR program. PIPA measurement of surface treatment effects provides a demonstrated ability to quantitatively measure initial treatment effectiveness along with the effect of operationally induced changes over the life of the treated component. Test specimens of steel and CMSX-4, a single crystal, nickel-based superalloy, were prepared and measured at incremental shot peening intensities ranging from 0-20A. The CMSX-4 samples were then subjected to a range of fatigue and thermal conditions to assess relaxation effects and to assess subsurface residual stresses. The PIPA technology proved to be highly sensitive to the induced changes in surface treatment intensity and accurately measured the relaxation of the shot peening intensity induced by varying degrees of simulated operational conditions. In addition, the tests demonstrated PIPA and related technologies' ability to quantify subsurface residual stresses in test specimens. Use of PIPA to nondestructively quantify surface and subsurface residual stresses in turbine engine materials and components, especially single crystal and complex geometry components, will lead to improvements in current, conservative engineering designs and maintenance procedures.

Introduction: Single crystal superalloys are used in a broad variety of applications in industry today, including critical turbine applications for power plants and commercial jet engines. Because of the high consequence potential of failure of these components, many are subjected to surface treatments that induce a compressive stress at the surface of the component to improve resistance to fatigue crack initiation and growth. Single crystal materials, such as nickel-based superalloys, provide increased fatigue and thermal resistance and extended life to turbine engine components, but due to their unique atomic structure, no nondestructive technology currently exists that can detect and accurately quantify surface and subsurface residual stresses/strains in these materials.[1] Many of the components manufactured from nickel-based superalloys are not only costly, but may have potentially catastrophic consequences if failure occurs.

Quantification of the effects of near-surface residual stresses induced by surface treatments and the ability to quantify the effect of interactive damage mechanisms that result in long term relaxation of the surface effects are critical issues in developing ways to improve the use of the nickel-based superalloys and other metal components. Unfortunately there is limited knowledge about surface treatment effects on operational components because only destructive methods are available for evaluation that prevent additional use of the component or only provide limited surface information on the effects (e.g., x-ray diffraction analysis). Shot peening and other surface treatments have been used for years as a means of inducing compressive residual stresses in a component's surface, which is considered to improve fatigue life. [2,3,4,5,6] Shot peening is considered effective at inducing surface residual stress in many metals to a depth of 300-400 micron depending on the hardness of the material. The intensity of the induced residual stress is typically quantified using the Almen intensity, which involves peening a strip of given dimensions and material (typically SAE1070 spring steel) with a given intensity and with a coverage of 200%. The Almen intensity is related to the shot velocity or pressure used to fire the peening pellets at the surface of the material. The Almen intensity is quantified by measuring the deflection in the arc of the strip caused by the change in subsurface residual stress distribution over a fixed length. However, this approach results in a number of problems as the Almen intensity is specific to the material type, stress profile and material hardness. Consequently, the Almen intensity level provides little information on materials other than the test material, and more specifically, on the effectiveness of shot peening on actual components.

Photon Induced Positron Annihilation (PIPA) and Distributed Source Photon Annihilation (DSPA) have demonstrated the capability to detect and quantify induced surface treatments and operational damage effects in single crystal materials. These technologies have the capability to vastly improve the understanding of microstructural states, failure mechanisms, and annealing treatments that can be used to improve critical component designs. The PIPA and DSPA technologies used in this research program were recently developed at the Idaho National Engineering and Environmental Laboratory (INEEL). The PIPA technology is a volumetric measurement technology that can measure microstructural damage at depths up to 4 inches in materials, whereas the DSPA technology uses high-energy positrons (nominally 3 MeV) that can be used to penetrate depths of up to about 2 mm in metals over large areas up to 100 cm^2. The DSPA process can be used to penetrate into greater depths in less dense materials such as plastics. These technologies are new additions to current material characterization technologies that when fully developed are expected to have specific applications to almost all materials industries. Specific improvements developed at INEEL and incorporated in this technology include the use of high-energy photons (15-20 MeV) to generate neutron deficient nuclei in materials (e.g., most metals, composites and polymers) that produce positrons within the bulk material for a few minutes to hours allowing bulk fatigue or lattice structure change/ damage to be accurately measured (<1% uncertainties). Other more portable approaches, based on neutron induced prompt gamma rays are under development.

The PIPA process generates positrons inside the bulk material through the application of high-energy X-ray bombardment of the target material by a linear accelerator. Positrons are formed when the X-ray interactions result in a photo-neutron reaction. The short-lived isotope decays into a more stable material through positron decay within a few minutes or hours. Positrons annihilate with electrons and produce gamma rays at 0.511 MeV with small momentum induced changes that are indicative of the quantity and type of defects present in the material. Positron annihilation occurs when a positron encounters an electron and their mass is converted into pure energy in the form of two gamma rays. If the positron and the electron with which it annihilates were both at rest or with little momentum at the time of decay, (i.e., in a defect), the two gamma rays would be emitted in exactly opposite directions (180 degrees apart), with an energy of 0.511 MeV, whereas if the annihilation occurs in a location without defects where electrons have momentum energy, the gamma ray measured has incremental, measurable differences from 0.511 MeV (Conservation of Energy). Figure 1 shows the PIPA process and Figure 2 depicts the formation and subsequent thermalization of the positron as it travels through the lattice structure, searching for a lower charge density region (defected area), where it becomes trapped and then annihilates with an electron.

The positrons created by the PIPA process are formed throughout the bulk material, achieving better sensitivity and accuracy of defect detection than historical surface positron beam spectroscopy. The depth of defect detection for PIPA is only limited by the attenuation of the annihilation gammas to be measured by the germanium detector; related to the material density. Additional information on the measurement processes used can be obtained from a number of references.[7,8,9,10,11,12,13]

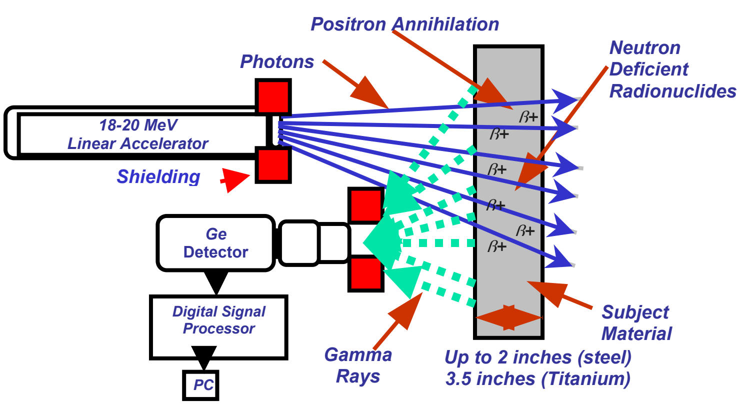

Figure 1. PIPA Process

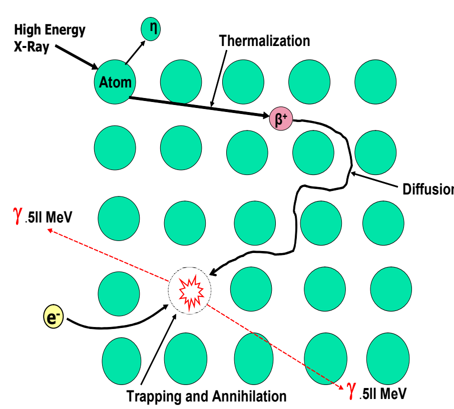

Figure 2. Positron Life Cycle

The primary measurement technique used is measuring the Doppler broadening of the 511 keV gamma ray peak. Although in principle, the Doppler broadened gamma ray spectrum can be deconvoluted to extract the electron momentum distribution, a simple shape parameter is commonly used to characterize the annihilation peak. Two parameters, S (for shape) and W (wings) are usually employed. The S-parameter is defined as the ratio of the counts (i.e., area) in a central region of the spectral peak to the total counts (area) in the peak, and W-parameter is defined as the ratio of counts in the wing region of the peak to the total counts in the peak.

Stress relief or relaxation of shot peening/surface treatment effects can occur due to thermal and/or thermomechanical effects. Characterization of the relaxation of compressive residual stresses has been limited because there is no nondestructive technology other than PIPA/DSPA that can be used to provide integral measurements of the stress relaxation effect for actual operational components; thereby providing an assessment tool, which allows current effectiveness and the need for a reapplication of the peening process to be evaluated. Shot peening, laser shock peening, and low plasticity burnishing represent some of the more effective surface treatments in use today on critical components. PIPA provides the capability to measure the effectiveness of these surface treatments and in addition provides the capability to measure subsurface residual stresses/strains up to10 cm into the component being examined.

Results: The evaluation of surface and subsurface residual stresses in the CMSX-4 single crystal, nickel-based superalloy was performed as a parametric study using the PIPA and DSPA technologies to evaluate the effects of incremental shot peening, temperature, and mechanical damage effects on both the surface and subsurface residual stress strain effects induced in the alloy test specimens. Test specimens were commercially prepared by MarTest, Inc and exposed to incremental shot peening intensities by Metal Improvement Company. Characterized samples with varying levels of induced residual stress were exposed to a parametric series of tests that varied thermal, fatigue and thermal-fatigue conditions.
For the CMSX-4 specimens, a test matrix was developed to parametrically test the four primary parameters being evaluated as part of this study. These parameters were 1) shot peening intensity, 2) thermal effects on surface/subsurface residual stress, 3) ambient temperature effects on surface/subsurface residual stress, 4) thermal-mechanical effects on surface and subsurface residual stress, and 5) variable load effects on surface/subsurface residual stress. The parameters and the range of the parameters tested were the following:

- *DSPA Surface/PIPA Volumetric Measurements* - Both DSPA and PIPA measurements were performed to assess the surface and subsurface residual stresses induced by the shot peening and the thermomechanical damage induced by the fatigue testing at several temperatures.
- *Shot Peening Intensity* - Shot peening intensities ranging from 2A through 20A with multiple samples at several levels were performed to allow direct comparison of the DSPA/PIPA responses at different temperatures and fatigue levels.
- *Thermal Time at Temperature Effects* **-** A time at temperature study was performed at 1000^{o}C for 100 and 500 hours to assess the effects of high temperature on low and high shot peening intensities.
- *Ambient Temperature Fatigue Damage* **-** Fatigue testing was performed at loads ranging from 4000 lbs (81,000 lbs/in^2) to 8000 lbs (163,000 lbs/in^2) for a 0.049 in^2 cross sectional area. Specimens with low and high shot peening intensities were subjected to tension-tension testing with R=1 (i.e., 100 lbs through 4100 lbs) for low (10,000) and higher (40,000) fatigue cycles which is considered representative of low cycle damage effects that might be expected for some turbine engine components.
- *Low and Medium Temperature Fatigue Tests* **-** Fatigue testing similar to that performed at ambient temperature was conducted at 300^{o}C and 700^{o}C. These temperatures were chosen, because the initial thermal tests indicate that the surface residual stresses relax quickly at higher temperatures. Consequently, these temperatures, which are lower than

turbine blade temperatures (1070°C) were used to characterize both surface and subsurface residual stress changes. Tests with lower (10,000) and higher (45,000) numbers of fatigue cycles were performed at loads of 4000 lbs (81,000 lbs/in^2), 6000 lbs (122,000lbs/in^2) and 8000 lbs (163,000lbs/in^2).

Near-Surface DSPA Measurement Results. Initial near-surface DSPA measurements were performed on the reference SAE1070 spring steel Almen strip samples and the CMSX-4 samples used for the thermal and fatigue tests, and volumetric PIPA measurements were performed on a selection of specimens at different shot peening intensities. The objectives of these measurements were to initially characterize all specimens to determine the DSPA and PIPA responses for the CMSX-4 material, and to assess the reproducibility of the shot peening process as measurements were performed on multiple samples with the same indicated shot peening intensity. Volumetric PIPA measurements were also performed on representative CMSX-4 specimens to assess the effect of surface compressive stresses on the PIPA response and to provide baseline data for measuring the effects of fatigue and thermal and mechanical damage on the CMSX-4 coupons.

Figure 3 and 4 show the monotonically increasing DSPA S parameter measurement response that corresponds to incremental shot peening intensities ranging from 0 to 20A for the initial Almen strip spring steel and CMSX-4 coupons, respectively. Figure 3 indicates that the DSPA S parameter response reaches a maximum at about 16A. This effect has been observed in prior tests on shot peened samples and is likely associated with the change in bead diameter at 16A from 230H to a larger bead size of 330H at 18A. There is a smaller reduction in response in 6A where the bead size changes from 110H to 230H.

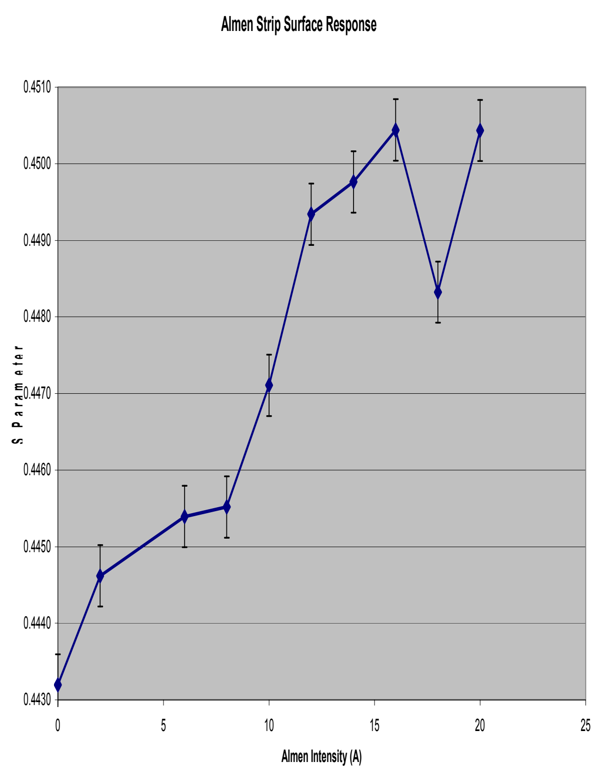

Figure 3. Almen Strip Calibration Response

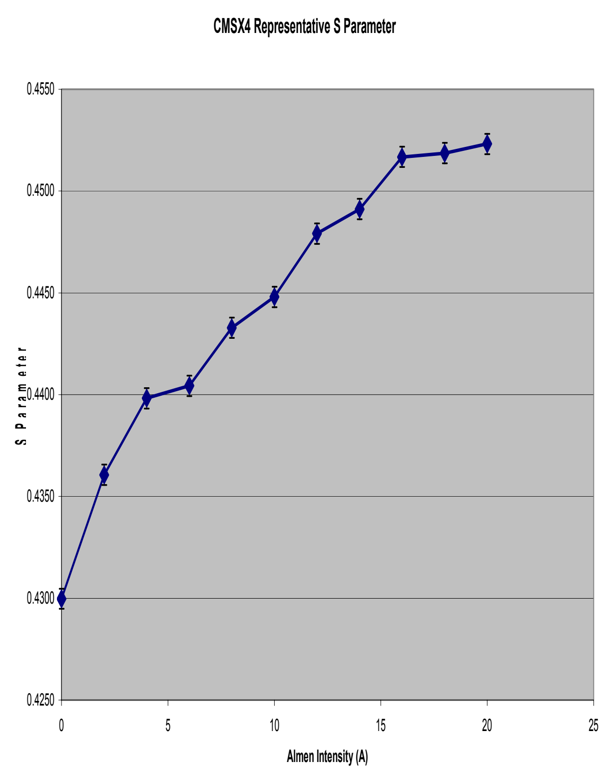

Figure 4. CMSX-4 Near-Surface DSPA Initial Characterization

The change in bead diameter during the shot peening process has been suspected of causing a reduction in the dislocation density in the material, caused by a change in the surface cold working characteristic differences induced by the different ball diameters. Comparison of the near-surface DSPA results for the two materials with the same shot peening intensities indicates that the dynamic range (from no shot peening through 20A) is 0.0223 for the CMSX-4 coupons, which is larger than that for the Almen strips (0.0055). These data indicate that the shot peening effect on the CMSX-4 material is likely greater than that for the SAE1070 spring steel and that DSPA measurements provide improved sensitivity across the measurement dynamic range of the CMSX-4 material.

Figure 5 shows the thermal and the combined thermal and fatigue testing effects on the surface residual stresses induced by shot peening in the CMSX-4 samples. The initial shot peening data are shown with 2 standard deviation or 95% confidence level error bounds that were calculated by a model that calculates the average error for a list of x,y values in a regression analysis. These bounds provide an estimate of the bounds for the undamaged surface residual stress condition. Also shown in Figure 5 are the near-surface DSPA measurement results for 1) thermal effects where the samples were heated to 1000°C for periods of 100 and 500 hours, 2) fatigue testing at 300°C, and 3) fatigue testing at 700°C. The data for fatigue tests performed at lower temperatures (<300°C) is not shown as it had little impact on the measured shot peening intensities with an average decrease in Almen strip reference intensity of 2A. At the higher temperatures (>700°C), the effects of the fatigue testing appear insignificant relative to thermal effects as the thermal conditions result in a significant relaxation of the subsurface residual stresses induced by the shot peening with S parameter values reduced to near or below the pre-shot peened condition. These data indicate that temperature appears to produce the most significant effect on shot peening intensity. The ability of the DSPA technique to measure the surface residual stress relaxation for the shot peening intensity under increased thermal environments provides the capability to assess the extent of relaxation induced in component surfaces by operational fatigue and thermal conditions.

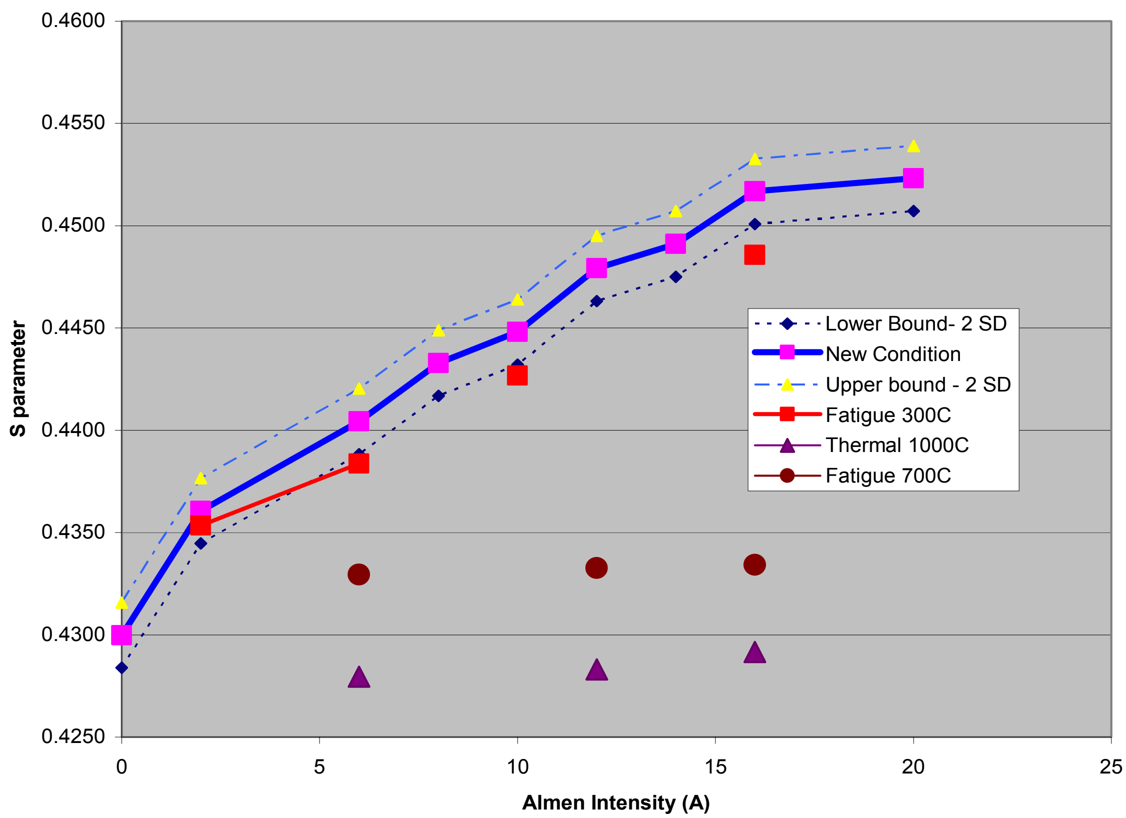

Figure 5. Thermal and Fatigue Effects as Compared to Original CMSX-4 Shot Peened Samples

Discussion: Summary results of an extensive evaluation of the effects of shot peening and the result of thermal and thermomechanical effects on the initial shot peening intensity are presented. Both the DSPA and PIPA processes have demonstrated the ability to characterize the surface and subsurface residual stresses induced in the CMSX-4 samples by shot peening, and the relaxation of these effects induced by the thermal and combined thermal and mechanical conditions to which the samples were exposed. Specific observations developed from this study are the following:

- Initial characterization of shot peened Almen strip test coupons made of SAE1070 spring steel with Almen intensities ranging from 2A to 20A clearly indicates the sensitivity of both DSPA and PIPA techniques to changes in the surface compressive residual stresses induced in the material due to changes in the shot peening bead size and increases in the applied impact air pressure.
- CMSX-4 coupons were shot peened to intensities ranging from 2A through 20A based on the Almen strip calibration process. An initial series of DSPA surface measurements were performed to assess the effectiveness of the shot peening process on the standard CMSX-4 test coupons and to assess reproducibility of both the shot peening process and the DSPA measurement process. Results of these measurements suggest that the CMSX-4 alloy is more affected by the shot peening process than is the SAE1070 spring steel. The measurements also indicate that the DSPA surface measurements at depths of 0.3 - 1 millimeter were more sensitive to the induced residual stress than the volumetric PIPA measurements.
- Thermal fatigue tests performed on the CMSX-4 specimens for 100 and 500 hours at 1000^{o}C indicate that this heat treatment results in complete recovery of the induced surface residual stress to the as-manufactured condition, as indicated by both the PIPA and DSPA S parameter measurement results. These results clearly show the capability of the PIPA technologies to quantify thermal effects (e.g., annealing) on surface residual and volumetric residual stresses.
- Combined thermal and fatigue testing of selected CMSX-4 test coupons with induced surface compressive residual stresses ranging from 2A to 18A was performed at ambient temperature, 300^{o}C, and 700^{o}C with a range of stresses ranging from the yield point down to about half the yield point (163/81 ksi) for the 300^{o}C and 700^{o}C temperatures. For a 0.049 in^2 specimen, the loads ranged from 4000 lbs to 8000 lbs, which is at or above the yield point depending on the temperature. The results of the surface DSPA measurements indicate that at ambient temperature, the induced surface residual stress was not affected by the fatigue damage until the yield stress was reached. Further, at the low temperature and number of fatigue cycles used, no correlation with the fatigue damage was observed in the surface measurement results. In contrast, the volumetric PIPA results showed a more direct correlation with the fatigue damage comparable with the ambient temperature measurements. The test results for the thermal and fatigue tests performed at 700^{o}C and 81 ksi load indicate that the subsurface residual stresses have clearly relaxed with no measurable compressive residual stress present. Both the near-surface DSPA and volumetric PIPA measurements indicate a quantifiable increase in the thermal-mechanical damage at this temperature that indicates that the higher temperature results in an increase in the buildup of damage in this temperature range relative to the ambient temperature tests.

Conclusions: The Almen strip and CMSX-4 coupon measurements indicate that the PIPA and DSPA technologies can successfully quantify surface and subsurface residual stresses induced by shot peening and likely all other surface treatments. Further, the results of this study clearly show the ability of the PIPA/DSPA technologies to quantify the impact of thermal and combined thermal and mechanical relaxation effects on the induced surface residual stress. The ability of the DSPA and PIPA technologies to quantify this effect has significant implications relative to improvements in the maintenance, surveillance and replacement of turbine engine components and in the improved safety of components using surface treatments.

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