D

automatic scanning. Also, the part can be determine the residual stress.

Surface stresses have been measured by surface (Rayleigh) waves. The disadvantage the acoustic velocity must be measured directly. Advantage of using surface waves is that they are not affected by the surface finish to those made by the well-established X-ray

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Materials Property Measurements

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EXPLORATORY RESEARCH
MATERIALS PROPERTY MEASUREMENTS

TASK 2: LIBRARY OF MEASUREMENT TECHNIQUES

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1.0 INTRODUCTION

The development of measurement techniques for characterizing the condition of materials for in-service or time-related aging is the primary objective of the Exploratory Research Project. Aging changes the mechanical properties of materials and, hence, the serviceability of the structures. The overall objective of the project will be an evaluation of various nondestructive measurements for determining the condition of materials as they age. The measurement techniques to be considered include ultrasonic velocity and attenuation, eddy current conductivity, neutron scattering and absorption, conventional and tomographic imaging for ultrasonic and radiation imaging, X-ray scattering, thermal impedance, and magnetic hysteresis.

This report summarizes an in-depth review of the measurement techniques that potentially could be used in materials characterization. The three sections of this report include a review of failure mechanisms in steel and a discussion of nondestructive evaluation (NDE) techniques and fracture mechanics, a description of a chart on Measurement Techniques versus Material Properties, and recommendations on the techniques and tests to be performed for the experimental investigations and analysis task of the project. As part of this task, a list of definitions for material characteristics has been compiled and listed in Appendix A. In order to understand the important materials properties to be measured, a review of the principles of fracture will be presented for background. Several measurement techniques have been reviewed to ascertain their potential to accurately estimate materials properties. The results of this review process have been put into a more manageable form for a better and more lucid overview of this complex topic. Specifically, a chart was chosen showing the measurement techniques and the materials properties that are measurable. Based on this information, five techniques have been selected and recommended for experimental verification and analysis. The selected material property measurement techniques are internal friction, acousto-elastic, eddy current, X-ray diffraction, and micro-indentation measurements. The plan would be to choose material samples stressed to induce changes in material properties (i.e., their dependence on dislocation density) and to perform a series of NDE measurements which will hopefully provide a unique correlation between them. These NDE techniques will be supplemented with metallographic measurements of grain size, dislocation density, and other measurements on materials properties as required.

2.0 FRACTURE REVIEW

Fracture is one of the properties of interest to the evaluation of in-service and time-related aging of materials. Through an understanding of the fracture mechanisms and related mechanical properties, the Materials Property Measurement Research Project will be striving to develop the capability to determine the materials condition of aged materials.

Fracture is not a simple process and can be affected by the temperature, grain size, environment, stress fields, and atomic structure. This section of the review will discuss the common forms of fracture; brittle and ductile. The presence or existence of a transition from ductile to brittle fracture will also be discussed. A summary of the two brittle fracture theories and the mechanical and microstructural aspects of the transition will be reviewed.

As previously mentioned, fracture is not a simple process; however in a fundamental understanding, there are only two requirements for fracture to occur. The first requirement is that of crack nucleation. Crack nucleation is the formation of the crack and is generally understood to occur by several different methods. The dislocation methods of crack nucleation are dislocation intersection, slip involving small angle tilt boundary, and dislocation alignment. Another nucleation mechanism involves a corrosive environment where chemical interactions form cracks. Once the cracks are formed, crack propagation must occur for fracture to proceed.

One experimental observation made by Petch [7] shows a relationship between the grain size and the fracture strength. His observations showed that as the grain size becomes smaller, the fracture stress increases. The empirical equation developed from this work is shown as equation (1).

$$\sigma_{\text{fracture}} = \sigma_0 + \frac{K}{\sqrt{L}} \quad (1)$$

where: σ_{fracture} = yield stress

σ_0 = yield stress with no grain boundary

K = parameter that determines effectiveness of grain boundary in raising yield stress

L = grain size (average)

The nucleation and propagation of a crack can be referred to in two manners, ductile and brittle fracture. Ductile fracture is the ability of a material to deform plastically prior to fracturing. Brittle fracture does not require plastic deformation; however, it can occur during the propagation of the crack. Ductile fracture is evident in a tensile test by the cup-cone appearance of the fracture surface. The process for this fracture is the nucleation and coalescence of voids to form a crack. The crack propagates until the tri-axial stress causes a large shear stress at approximately 45° to the axis and fracture occurs. Two typical stress-strain curves for ductile

fracture are shown in Figures 1 and 2. A brittle fracture stress strain curve is shown in Figure 3. (NOTE: Figures 1-3 are taken from Metals Handbook, eds. H. E. Boyer and T. L. Gall, ASM, 1985, p. 2-18).

FIGURE 1. Typical Ductile Stress Strain Curve

FIGURE 2. Typical Ductile Stress Strain Curve for Materials Showing Upper and Lower Yield Points

FIGURE 3. Typical Brittle Stress Strain Curve

The fact that a ductile-to-brittle transition can occur is most easily observed from experimental data such as the fracture toughness tests on steels with varying amounts of carbon. Figure 4 shows data from a Charpy Impact Test for carbon contents from 0.01% to 0.67%.

FIGURE 4. Charpy Impact Test Data Showing the Ductile-to-Brittle Transition (ref. [7], McClintock and Argon, p. 561).

A detailed study by Hahn [7] also shows a complex transition in mild steel and would be typical of all materials showing an upper and lower yield point. Figure 5 shows the results of Hahn's testing.

The classical brittle fracture theory developed by Griffith [7] assumed that a preexisting crack was present and that for fracture to occur the surface energy to open the crack had to be exceeded. The Griffith equation is given as:

$$\sigma = \left(\frac{2E\gamma_s}{\pi c} \right)^{1/2} \quad (2)$$

where: σ = fracture stress,
 E = modulus of elasticity,
 γ_s = surface energy and plastic work, and
 c = half the crack length.

This work was extended by Inglis [7] to include the effect of a finite crack width and included the effect of the crack tip radius:

$$\sigma_{\max} = \sigma_0 (1 + 2\sqrt{c/\rho})$$

where: c = half crack length,
 $\sigma_{\max}$ = maximum stress at crack tip,
 σ_0 = applied stress, and
 ρ = crack tip radius.

FIGURE 5. Data by Hahn, et al. Showing Complex Transition in Mild Steel Versus Temperature (ref. [7], McClintock and Argon, p. 518).

One problem with this theory was that it only included the surface energy term. With the development of dislocation theory came a new brittle fracture model.

Cottrell [19] developed a model for brittle fracture that includes the effect of dislocations, Petch's grain size effect, and friction stress (back stress of a dislocation). From dislocation theory:

$$2\gamma' = \sigma nb \quad (4)$$

where: γ' = effective surface energy including plastic work term,
 σ = applied stress,
 n = number of dislocations, and
 b = Burgers vector.

Cottrell's general equation for fracture stress is:

$$\sigma = \left[\frac{\gamma' G}{\pi(1-\nu)c} \right]^{1/2} \quad (5)$$

where: G = shear modulus,
 ν = Poisson ratio,
 γ' = effect of surface energy including plastic work term, and
 c = half crack length.

Cottrell's theory was developed from friction stress and Petch's equation and gave an equation for the yield stress.

In order to minimize the effect of the transition from ductile to brittle fracture, several chemical and microstructural modifications can be made. For example, the operating temperature can be defined for the use of the material. Chemical type adjustments such as the addition of interstitials, solution hardening, and precipitation aging are possible to form pinning sites for the dislocations. The principle of chemical type adjustments is based on the assumption that the stress to move a dislocation is proportional to the inverse of the dislocation length. Radiation treatment and grain size can also change the transition temperature. Quenching of the steel can be performed to lock in vacancies, which also affects the transition temperature. These are a few of the processing steps that can be taken to control the ductile/brittle transition temperature.

3.0 FRACTURE MECHANICS AND NDE

The development of an accept/reject criteria for materials and structures requires the coupling of nondestructive evaluation with fracture mechanics and structural analysis. As an example of the need for this coupling, it was found that the combined errors of underestimated crack growth rates and overconfidence in the inspection methods resulted in the crash of an F-111A in 1969 [1].

In order to use the results obtained from NDE in a fracture analysis, it is necessary to develop a methodology which incorporates the advantages and disadvantages of the two disciplines. The fundamentals of fracture need to be categorized and understood within the limits of what NDE measurements can be made. Simultaneously, the techniques and resolution of the various NDE methods have to be quantified to satisfy the fracture analysis requirements.

Reference to the coupling of the two techniques can be found in both NDE and fracture literature [2,3,4]. In the opening comments of the July 1979 meeting on Quantitative NDE, Dr. Donald Thompson gave the following definition of NDE:

Nondestructive evaluation (NDE) represents the capability to assess the state of a material, a component, or a structure from a series of quantitative and nondestructive measurements, and to predict the remaining serviceability of the item in question from the measurements and their evaluation in the context of appropriate failure models [5].

This definition is aimed at ensuring that NDE and fracture mechanics are joined to develop reliable accept/reject criteria.

The separation of a body into two parts is commonly referred to as fracture. A further breakdown of fracture is made by the designation of brittle, ductile, and transition (brittle to ductile) fracture. Brittle fracture is the rapid propagation of a crack with no plastic deformation. Ductile fracture is a slower process with extensive plastic deformation (similar to material stretching). Transition fracture primarily refers to body-centered cubic metals, such as iron, which fail by either brittle or ductile fracture depending on the temperature or strain rate [6,7].

As previously discussed, NDE provides an assessment of the state of a material. In order to characterize the state, some of the more common NDE techniques used are visual, radiation, ultrasonic, eddy current, magnetic measurements, acoustic emission, and optical and acoustic holography. Quantitative measurements are essential for the use of NDE data in a fracture analysis. Developments in quantitative measurements for the NDE techniques are being developed. The main objective of the qualitative research is to measure the size, shape, location, orientation, and materials properties of flaws for use in fracture mechanics analysis.

The NDE equipment includes the general and advanced NDE systems described in this report. The fracture mechanics analysis included Finite Element analysis computer programs and mechanical testing equipment such as tensile, compressive, and torsional test instruments.

4.0 MEASUREMENT TECHNIQUES

A Materials Property Measurement workshop as part of the project was held on February 9, 1987. The following measurement techniques were discussed: internal friction, magnetic hysteresis, dye penetrant, magnetic particle, eddy currents, thermography, optical holography, Auger electron spectroscopy, X-ray and ultraviolet photo-emission spectroscopy, Mossbauer spectroscopy, Low/High energy electron diffraction, X-ray diffraction, neutron diffraction, optical microscopy, TEM and SEM microscopy, photoacoustic microscopy, ultrasonics and acousto-elastics, inversion techniques, acoustic emission, and hardness testing.

A chart of these techniques for measuring materials properties has been generated. Appendix B lists the references for the NDE Materials Characterization Chart. The properties include mechanical, electrical, magnetic, ageing, hardness, texture, dislocation density, etc.

From the workshop it was determined that multiple measurement techniques will be required to develop a complete characterization of the material system being investigated. A simple metal system was suggested to minimize the complexity of the governing fracture mechanism and, therefore, a low-alloy steel was suggested.

The measurement techniques showing the most promise from the initial review are internal friction, ultrasonics and related acousto-elastic properties, eddy current, and hardness measurements. These will be supplemented with the material science methods of microscopy, and X-ray and neutron diffraction. Other techniques such as auger, electron, and Mossbauer spectroscopy will be used as required.

A brief summary of the recommended measurements to be performed for the experimental investigations and analysis task of the project will be given. Papers on the specific topics shown in the Materials Properties Chart have been collected and are filed in PSL Room 1401 for easy reference. Appendix C is a copy of the February Monthly Report containing the minutes from the workshop and copies of the presentation materials on materials characterization techniques.

4.1 INTERNAL FRICTION

Internal friction measurements involve the determination of atom movements within solids. Some of the mechanisms leading to damping (internal friction) at low stress levels are thermal currents, stress diffusion of solute atoms, grain boundary sliding, and simple dislocation interactions [7,8]. Solids are never perfectly elastic, and some of the propagating elastic wave energy is lost through conversion to heat. The various mechanisms by which this conversion process occurs are collectively called internal friction (as differentiated from the derivation below based solely on interstitial mechanisms). The conversion mechanisms include the following: relaxation by thermal diffusion, atomic diffusion, magnetic diffusion, ordered distributions, preferential distributions, stress along previously formed slip bands, stress across grain boundaries, and stress across twin interfaces [7]. Internal friction is used to test authenticity of coins, the soundness of castings, and the quality of musical instruments and glassware.

An analogy for internal friction is the damping, or energy loss, in an electrical system. The evolution of the term is from the study of interstitial diffusion that makes use of the Snoek effect. If a stress is applied, two types of strain will occur: the normal elastic strain and the anelastic strain (which is caused by the movement of solute atoms into sites with axis parallel to the stress axis). Therefore, when a stress level is changed, the elastic part will track the strain; the anelastic part will be delayed and follow a relaxation time that is a function of the material properties. This phenomena is the Snoek effect [7].

The conventional and most frequent way that the relaxation times of materials are measured is with a torsion pendulum. Once the torsion pendulum is set in motion, its decay will follow that of a damped sinusoidal vibration. The damped sinusoidal is a monotonically decreasing function cycle-to-cycle.

Let A1 and A2 be the peak-to-peak amplitudes of two consecutive cycles with A1 occurring in time before A2. The energy of vibration in an oscillating system is proportional to the square of the vibration amplitude. If E is the energy of the pendulum and @E is the frictional loss of energy per cycle, then:

$$@E/E = [(A2)^2 - (A1)^2]/(A1)^2 \quad (6)$$

This equation is normally called the specific damping capacity.

Because of the anelastic strain component, the strain lags the stress by the phase angle α . The tangent of this phase angle can also be used as an index of the internal energy loss.

$$1/Q = \text{tangent } \alpha = 1/\pi * \ln(A1/A2) \quad (7)$$

This quantity is called the internal friction.

Internal friction damping is maximum when the frequency used in an acoustic measurement is near to that defined by the equation:

$$f = \frac{\pi}{2} \left(\frac{D}{h^2} \right) \quad (8)$$

where D is the thermal diffusion coefficient and h is the specimen thickness.

The macroscopic development of internal friction damping is a relaxation process. The macroscopic evaluation determines the motion of dislocations that are pinned by impurities, precipitates, and other dislocations and is based on the mechanical behavior of grain boundary slip for crystalline materials.

The measurement of internal friction has been performed with acoustic methods using frequencies between 20 and 20,000 Hz. Early work on internal friction goes back to 1937 by Zener. Other NDT experts publishing work on internal friction include: Wolfgang Sachse, Bob Green, and Don Thompson. Papers on internal friction can be obtained from the Materials Property Measurement files set up for this project.

3.2 ACOUSTO-ELASTIC MEASUREMENTS

Ultrasonic measurements can be used for determining the elastic properties of materials [9,10,11,12]. Auld in Acoustic Fields and Waves in Solids lists the Christoffel equations for isotropic and anisotropic solids. By using these relationships, wave velocity surfaces may be generated and are a function of a material's elastic moduli or compliance matrix. Therefore, a material's elastic constants may be determined from ultrasonic velocity measurements. For isotropic materials the longitudinal and shear wave velocities are:

$$v_L = \left(\frac{C_{11}}{\rho} \right)^{1/2} \quad (9)$$

$$V_s = \left(\frac{C_{44}}{\rho} \right)^{1/2} \quad (10)$$

where: C_{11}, C_{44} = elastic constants
 ρ = density

Other crystallographic symmetries such as cubic, hexagonal, trigonal etc. require the determination of up to seven independent elastic constants.

Ultrasonics can also be used to determine the surface stress or bulk stress of a solid. In many cases, it is necessary to have a sample of the parent material for calibration.

The ultrasonic measurement of stresses is possible because of the acousto-elastic effect. The acousto-elastic effect is the nearly linear change in acoustic velocity with stress. Stress measurements have been made using longitudinal, shear, and surface (Rayleigh) waves.

The relationship between stress and strain is usually considered to be a linear function as expressed by Hooke's Law [9]:

$$\sigma = M\epsilon \quad (11)$$

where: σ = stress,
 ϵ = strain, and
 M = elastic modulus.

However, materials deviate slightly from this idealized behavior. This deviation has been called the anharmonic property of a solid, and it can be expressed as a power series [9]:

$$\sigma = M\epsilon + C\epsilon^2 + D\epsilon^3 + \dots \quad (12)$$

where C is the third order anharmonic constant, D is the fourth order anharmonic constant, and so on.

If just two terms of the power series are considered, the stress strain relationship can be written as [9]:

$$\sigma = M'\epsilon$$

where: $M' = M + C\epsilon$

The velocity of sound is related to the material properties by the expression [9]:

$$V = \left[\frac{M'}{\rho} \right]^{1/2} \quad (13)$$

where V is the velocity of sound and ρ is the density.

Differentiating the velocity equation gives [9]:

$$dV = \frac{V}{2} \left(\frac{dM'}{M'} - \frac{d\rho}{\rho} \right) \quad (14)$$

From the above equation, it can be seen that stress affects the velocity of sound because of the anharmonic property of the solid and because stress changes the material density for materials with a Poisson's ratio less than 0.5.

Polarized shear waves have been used by Fukuoka, Toda, and Yamane [12] to measure the bulk residual stress in a patch-welded disk using shear-wave birefringence. The amount of birefringence (i.e., the difference in velocity of the two polarized shear wave components) is related to the principal stresses by the equation [12]:

$$V_{T1} - V_{T2} = \frac{V_2}{2\mu} \left(1 + \frac{C}{\mu} \right) (\sigma_1 - \sigma_2) \quad (15)$$

where: V_{T1} and V_{T2} = the acoustic velocities of the shear waves polarized in the principal directions,
 V_2 = the shear wave acoustic velocity in the non-stressed isotropic material,
 μ = the modulus of rigidity,
 C = the third order anharmonic constant, and
 σ_1 and σ_2 = the principal stresses.

In the study, it was necessary to measure the constant relating the acoustic velocities and principal stresses by making measurements of uniaxially loaded specimens made from the parent material. It was also necessary to control the sample temperature closely.

The acoustic velocities were measured by the so-called sing-around method where the output from the receiving transducer is used as a trigger for the sending transducer, thus producing a succession of pulses. The frequency of the triggering is used to calculate the acoustic velocity. The sing-around technique provides a significant improvement over direct measurement of the time of flight.

The major disadvantages of using the shear-wave birefringence method are that the transducer must be attached directly to the sample and the transducer must be rotated to change polarization.

Longitudinal waves are used to measure bulk stress in much the same way as shear waves. The velocities can be measured either by using the sing-around technique or by monitoring the frequency shifts necessary to null the acoustic pulses reflected separately from the front and back faces of the test piece. However, using longitudinal waves provides several advantages over shear-wave birefringence. First, the probe and sample may be immersed in water eliminating the need for physical attachment of the probes to the sample and allowing

automatic scanning. Also, the part can be inspected at multiple angles to determine the residual stress.

Surface stresses have been measured by Jassby and Saltoun [11] using surface (Rayleigh) waves. The disadvantage of using surface waves is that the acoustic velocity must be measured directly by time of flight. The advantage of using surface waves is that the stress measurements can be compared to those made by the well-established X-ray diffraction technique. Also, the stresses within a solid can vary significantly with depth, thus making the measurement of bulk stress using shear and longitudinal waves somewhat artificial. The surface stress can also be correlated to the critical reflection angle.

All of the aforementioned ultrasonic stress measurement techniques have one disadvantage; e.g., it is necessary to make calibration tests on a sample made from the parent material. Several reasons exist for this. First, the anharmonic constant data for many materials is not widely available. Second and more importantly, there are many material characteristics that affect the ultrasonic velocity to the same degree as stress. These include temperature, micro-stress, multiple phases, and composition gradients.

Measurements are typically made using relatively small ultrasonic probes. Special circuitry may be required to determine the ultrasonic velocity with great precision.

4.3 EDDY CURRENT MEASUREMENTS

The use of eddy current probes and systems has been used for the characterization of residual stress, yield stress, and hardness [13,14,15,16]. In eddy current testing, the material properties affecting the test data include: electrical conductivity, magnetic permeability, temperature, property depth, and surface conditions.

The studies on residual stress have been performed on low-alloy pressure vessel steel (ASTM A533) and heavy-section forging steel (ASTM 471) [13,14]. Excellent correlation between the eddy current data and stress was achieved. Stress contour maps have been successfully generated on simple as well as complex stress specimens [15]. Test results have shown the importance of calibration standards for reliable data interpretation. Recent work on eddy current determination of hardness has shown excellent correlation with case wall hardness of brass.

4.4 MICRO-HARDNESS MEASUREMENTS

A micro-hardness technique has been developed for material property characterization. F. Haggag [17] at Battelle Columbus Laboratory has fully automated a micro-indentation tester using Corporate Technology Development funds. A test is performed using a hydraulic test machine that is computer controlled. Load and indentation depth data are collected during the test using a data acquisition system. The yield stress could be determined using empirical correlations. The amount and geometry of the material pile-up around the final indentation could be used to estimate the residual stress in a structure.

As part of PNL's test program, the micro-indentation system at Columbus Labs will be utilized as part of the technical exchange on Material Property Characterization.

4.5 X-RAY DIFFRACTION

X-ray diffraction (XRD) can be used to determine the surface stress of an isotropic crystalline solid [9,10,18]. Measurements are limited to surface depths less than 0.013 mm. It is not necessary to make measurements in the unstressed state.

X-ray diffraction allows the surface stress in a particular direction to be calculated based on information from the X-ray diffraction pattern.

Figure 6 shows a sample with atomic planes spaced d apart and illuminated by coherent X-rays at angle θ . When the difference in the path lengths of the X-rays reflected from the two atomic planes is an integer multiple of the wavelength ($n\lambda = L_1 + L_2 - L_3$), constructive interference occurs at the observation point. When L_1 , L_2 , and L_3 are written in terms of d and θ , the result is Bragg's Law:

$$n\lambda = 2d \sin \theta \quad (16)$$

FIGURE 6. Reflection of X-rays from Atomic Lattice Planes of Surface

Thus, the spacing between the atomic planes can be determined by measuring the angle θ when the wavelength is known. From Bragg's Law, it is clear that λ must be less than $2d$ for an interference pattern to be produced. X-ray interference peaks with a θ angle of near 90° are preferred because they are the most sensitive to residual stress.

A typical arrangement for the measurement of residual stress in the direction f using the double-exposure or two-angle technique is shown in Figure 7. Here, d_n is the spacing of the atomic planes that are parallel to the surface, and d_i is the spacing of the atomic planes whose normal vector makes an angle ψ with the surface-normal vector as shown. The strains in the surface normal and ψ directions, respectively, are [10]:

$$\epsilon_3 = \frac{d_n - d_0}{d_0} \quad (17)$$

and

$$\epsilon_\psi = \frac{d_i - d_0}{d_0} \quad (18)$$

where d_n is the spacing of the atomic planes parallel to the surface, d_i is the spacing of the atomic planes normal to ψ , and d_0 is the spacing of the unstressed atomic planes.

FIGURE 7. Surface Stresses (ref. [10], Boyd and Shackelford, p. 8)

In an isotropic solid, the strains ϵ_3 and ϵ_ψ are related to the surface stress in the f direction by the relationship [10]:

$$\epsilon_\psi - \epsilon_3 = \frac{\sigma_\phi}{E} (1-\nu) \sin^2 \psi \quad (19)$$

where E and ν are Young's modulus and Poisson's ratio, respectively.

Since $d_i \approx d_n \approx d_0$ (small strain approximation), σ_ϕ can be written as [10]:

$$\sigma_\phi = \frac{E}{(1 + \nu) \sin^2 \psi} \left[\frac{d_i - d_0}{d_n} \right] \quad (20)$$

The two-angle technique allows the direct measurement of the diffraction angle θ so it is more convenient to put the expression σ_ϕ in the following form [10]:

$$\sigma_\phi = \frac{E}{(1 + \nu) \sin^2 \psi} \left[\frac{\sin \theta_n}{\sin \theta_i} - 1 \right] \quad (21)$$

where θ_n is the diffraction angle for the atomic planes that are parallel to the surface, and θ_i is the diffraction angle for the atomic planes whose normal vector is at an angle ψ .

A refinement of the two-angle technique is the multi-angle or sine-square-psi technique. Using this technique, θ is measured at several ψ angles, and the residual stress σ_ϕ is obtained from the slope of the linear plot of ϵ_ψ versus $\sin^2 \psi$.

5.0 RECOMMENDATIONS ON EXPERIMENTAL INVESTIGATIONS TASK

As discussed in the review on fracture, the grain size and dislocation density have been shown through the Petch [7] relation and Cottrell [19] models to correlate to the fracture stress. From this review, it was determined to chose material characterization techniques best suited to measuring these properties.

The review of nondestructive techniques for characterization of materials measurement has identified the importance of multiple measurements. The recommended techniques to be used for the initial phases of the experimental investigations and analysis task of the Exploratory Research Project are internal friction, acousto-elastic, eddy current, X-ray diffraction, and micro-hardness measurements. These techniques will be supplemented with materials measurement methods for determining grain size and dislocation density of samples.

The materials systems to be investigated are a high-purity 304L stainless steel and/or high-purity nickel. Tensile test samples will be fabricated. Three different grain sizes will be annealed into the test sample lots. The samples will then be strained to several predetermined stress levels in order to induce an increasing level of dislocation density into the samples. The different materials characterization methods described above will then be used to evaluate the test samples. Test result data will be evaluated for correlation to the dislocation density and grain size in the samples.

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APPENDIX A

Material Characteristics Definitions

Bulk Modulus [B, pp. 191-192] Hydrostatic compression introduces a volume contraction which is initially proportional to pressure. The bulk modulus is the ratio of the hydrostatic pressure to the resulting volume contraction. Young's modulus and the bulk modulus are related through Poisson's ratio.

Creep/Creep Rate [A, pp. 625-650] Creep is the time-dependent inelastic deformation of a solid under applied stress. Creep rate is a function of temperature. Three stages of creep can often be distinguished: primary creep at a decreasing rate under constant stress, secondary creep at a more or less constant rate, and tertiary creep at an accelerating rate followed by fracture. Some of the processes leading to secondary creep may be (a) the climb of edge dislocations out of slip planes to overcome obstacles; (b) drag of sessile jogs on screw dislocations; (c) elimination of dislocation loops around precipitates; (d) grain-boundary sliding; (e) accommodation mechanisms, such as grain-boundary migration, which are required for strain compatibility in grain-boundary migration; and (f) migration of vacancies between grain-boundaries.

Density - Density is the ratio of mass to the volume occupied.

Dislocation Density [A, pp. 96-145] Dislocations are faults in the stacking of the atoms making up the crystal. Plastic deformation in crystalline materials is accomplished by the generation and motion of dislocations. The dislocation density is the number of dislocations in a given space.

Ductility [A, pp. 325-326] The ability of a material to undergo plastic strain before fracture is called ductility. One measure of ductility is the reduction in area (necking) of a tensile test sample at fracture, and another measure is the elongation defined as a change in gauge length to fracture divided by the original length of a tensile test sample. Plastic deformation in crystalline materials is accomplished by the generation and motion of dislocations.

Electrical Conductivity [B, pp. 283,288] In conductors, the active charge carriers are free electrons. The limiting factor for conductivity in metals is the mobility of the electrons, which is determined by the mean free path. The mean free path is the average distance between scattering points (irregularities in the crystal structure) in the electron path. Electrical conductivity is affected by solution hardening, strain-hardening, and temperature.

Energy-Transition (Fracture Transition) Temperature [A, pp. 557-565] Body-centered cubic metals such as iron and tungsten experience a discontinuous decrease in ductility as temperature is decreased. The temperature at which this discontinuity occurs is called the energy-transition temperature. Below the energy-transition temperature the metal experiences brittle fracture and above this temperature it experiences ductile fracture. Several factors that influence the energy-transition temperature are grain size, strain rate, impurities, and radiation exposure. Face-centered cubic metals such as stainless steels do not exhibit fracture-mode transition.

Fatigue [A, pp. 576,612] Fatigue is progressive fracture caused by repeated loading. At a high enough strain level, repeated slip produces cracks, usually in slip bands at the surface or at an interface with an inclusion, within 1% to 30% of the fatigue life. Such cracks then grow to fracture, either through metallurgical damage or progressive deformation at the crack tip. Fatigue tends to be controlled by local rather than nominal stress. Fatigue life is affected by surface finish, corrosion, hardness, temperature, inclusions, and structural details.

Ferromagnetism [B, pp. 327-332] Iron and related materials such as nickel and cobalt can possess a very high magnetic permeability at temperatures up to their Curie temperature. The atoms develop a coupling whereby they spontaneously align their magnetic moments with one another to produce a magnetic domain. Each domain acts as a magnet. In "soft" magnetic materials in the absence of an external magnetic field, there are as many domains oriented in one direction as another, so the magnetism is not obvious. In "hard" magnetic materials, permanent magnets, the number of domains having one orientation exceeds the number having other orientations, so a net magnetization exists. Any factor which interferes with the movement of domain walls adds to the permanency of a magnet. Non-magnetic inclusions and dislocations are effective in this respect. In fact, this relationship accounts for the origin of the terms "hard" and "soft" to describe the permanency of magnets.

Fracture Stress - The fracture stress is the stress at which a material sample fails during tensile testing.

Grain Size [C, p. 19] Grain size is a measure of the areas or volumes of the grains in a polycrystalline material.

Lame Constants [A, pp. 80-81] The Lame constants, typically called λ and μ , are material constants that are related to the shear and dilational wave speeds.

Magnetic Permeability [B, pp. 324-337] The magnetic permeability of a material is the ratio of the magnetic flux density to the applied magnetic field. The permeability is often expressed as the relative permeability which is the material's permeability divided by the permeability of a vacuum. The permeability of a material can also be described by its magnetization or magnetic susceptibility.

Phase [C, p. 27] [A, p. 159] A phase is a homogeneous and distinct portion of a material system such as carbide precipitates in an iron matrix.

Poisson's Ratio [B, pp. 190-191] Lateral dimensional changes accompany axial strains. The ratio of lateral strain to axial strain under the influence of uniaxial loading is called Poisson's ratio. Values range from 0.25 for glass and hardened steel to 0.5 for soft rubber and other materials which maintain constant volume when stressed elastically.

Precipitation Hardening [A, pp. 157-159] Precipitation hardening is a two-step process. First, a supersaturated solid solution is obtained by quenching from an elevated temperature. Then the metal is aged at an intermediate temperature allowing the solute to precipitate. The result is a uniformly dis-

persed collection of closely spaced small precipitates. Such precipitates may produce hardening the same way the solute atoms do, and they may prevent dislocations from passing through them by a high hardness of their own or because their lattices are not lined up with the bulk material lattice. The result is that dislocations are forced to extrude through the free spaces between precipitates, thus producing resistance to dislocation motion (hardening).

Quench-Hardening [A, pp. 159-160] If a metal is raised to a temperature near its melting point, a relatively large vacancy concentration can be held in thermal equilibrium. After rapid quenching, the vacancies condense into vacancy clusters or disks. When large enough, the vacancy disks collapse to form sessile dislocation rings. Sessile dislocation rings are strong obstacles to dislocation motion.

Radiation Hardening (Aging) [A, p. 159] Irradiation of metals with charged or uncharged heavy particles produces vacancy clusters or disks inside the material. When the radiation is made up of alpha particles, appreciable quantities of helium gas can also be entrapped in the metal. Nuclear reactions may produce similar gas entrapment. Vacancy disks, when large enough, collapse to form sessile dislocation rings. The sessile dislocation rings that form are strong obstacles to dislocation motion.

Remanent Induction [B, p. 328] Remanent induction is the magnetic induction that remains in a permanent magnet when the applied magnetic field is removed.

Saturation Induction [B, pp. 328-332] The net magnetization of a ferromagnetic material can be increased if an external magnetic field is applied. Those domains which are favorably oriented grow at the expense of those which are not oriented with the field. With domain growth, the net flux density increases rapidly until most of the domains are aligned in the same direction at which time the flux density increases very slowly with an increasing magnetic field. This is called saturation induction.

Shear Modulus [B, pp. 192-194] The shear modulus is the ratio of shear stress to the resulting elastic shear strain. Young's modulus and the shear modulus are related through Poisson's ratio.

Solution Hardening [A, pp. 153-157] Metal may be hardened by alloying it with small amounts of soluble additions. The soluble additions appear as separate foreign atoms in the crystal and produce a misfit by dilation or by distortion of the lattice around the addition. The resulting stress fields exert forces on nearby dislocations.

Surface Emissivity - Surface emissivity is the efficiency with which a material radiates thermal energy as photons.

Strain-Hardening (Work-Hardening) [A, pp. 160-190] Strain-hardening is the decrease in the motion or generation of dislocations due to plastic strain.

Temperature - Temperature is the measure of thermal energy.

Texture [C, p. 38] In a polycrystalline aggregate, the texture is the state of distribution of crystal orientations. In the usual sense, it is synonymous with preferred grain orientation as is characteristic of plastically deformed metals.

Thermal Capacitance [B, p. 290] Thermal capacitance is the ability of a material to store thermal energy. In metals the thermal energy is stored as the kinetic energy of atoms and free electrons.

Thermal Conductivity [B, pp. 288-290] In metals thermal energy is propagated by means of phonons (quantized elastic waves) and electrons. Since electrons move readily through a metal, a large fraction of the thermal energy is transferred by electrons, and therefore a direct comparison can be made between thermal and electrical conductivity. Like the electrical conductivity, the thermal conductivity of a metal is affected by solution hardening, strain-hardening, and temperature.

Toughness [B, p. 7] [A, pp. 567-570] Toughness is the energy required for fracture. It is represented as the amount of energy required to break a test specimen in, for instance, a Charpy V-notched impact test.

Ultimate Tensile Strength [A, pp. 7-8] The ultimate tensile strength or tensile strength is the maximum load per unit original area obtained when tensile testing a material sample.

Yield Strength [A, p. 7] The yield strength is the stress required to produce some specified plastic deformation, usually of the order of a few tenths of one percent. It is used for design purposes to approximate the maximum stress without plastic deformation.

Young's Modulus [B, pp. 188-190] Young's modulus is the ratio of uniaxial stress to the resulting strain. The values of Young's modulus are directly related to the interatomic bonding energies. Materials with deep and narrow energy troughs have a high Young's modulus.

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APPENDIX B

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TECHNIQUE PROPERTY	SEMI-DESTRUCTIVE METHODS		SPECTROSCOPIC METHODS		THE MET
	HOLE & RING DRILLING	MICRO-INDENTATION	MOSSBAUER SPECTROSCOPY [24]	X-RAY/ULTRAVIOLET PHOTOEMISSION SPECTROSCOPY [21]	THERMOGRAPHY
RADIATION HARDENING			MAYBE	MAYBE	
SOLUTION HARDENING			MAYBE	MAYBE	
PRECIPITATION HARDENING			YES [24]	MAYBE	
QUENCH HARDENING			MAYBE	MAYBE	
ENERGY-TRANSITION TEMPERATURE					
DUCTILITY			MAYBE	MAYBE	MAYBE
DISLOCATION DENSITY					
STRAIN HARDENING					
TOUGHNESS [48,43]		YES			
HARDNESS [31]		YES [44]			
CREEP/ CREEP RATE		YES			MAYBE
FATIGUE					
FRACTURE STRESS					
YIELD STRENGTH [43]		YES			YES
ULTIMATE TENSILE STRENGTH [43]		YES			
APPLIED STRESS	YES [38,4]	YES [5]			YES
RESIDUAL STRESS [38]	YES [38,4]	YES [5]			YES
TEMPERATURE					YES
THERMAL DIFFUSIVITY					
SURFACE EMISSIVITY					
THERMAL CAPACITANCE					
COEFFICIENT OF THERMAL EXPANSION					
ELECTRICAL CONDUCTIVITY					

[X] IN

[illegible]

EASUREMENT METHODS

[X] INDICATES REFERENCE FOUND IN APPENDIX B OF LIBRARY OF MEASUREMENT TECHNIQUES REPORT, BOYD, GREEN, DOCTOR, GOOD

ACOUSTIC METHODS							
CRITICAL ANGLE	INTERNAL FRICTION [11,42,44]	OPTICAL HOLOGRAPHIC INTERFEROMETRY	ACOUSTIC MICROSCOPY [40]	INVERSION OF SCATTERING DATA [4,12,16,32,25,33,34]	SYNTHETIC APERTURE FOCUSING TECHNIQUE	ULTRASONIC TOMOGRAPHY	ACOUSTIC ELASTICITY MEASUREMENTS [
	YES						
	YES [42]						Y [
	YES	MAYBE					
	YES						M/
	YES [43,44]	MAYBE					M/
MAYBE [31]	YES [44]	YES	YES			YES	[1
	YES	YES					
		YES					
	MAYBE [43,44]	YES					
	YES [44]	YES					
YES [5]	YES [5]	YES	YES [5]			YES	[20,4
YES [5]	YES [5]	YES	YES [5]			YES	[20,4
	YES	YES	YES			YES [29]	
	YES	MAYBE					
	YES						

CONDUCTIVITY					
MAGNETIC PERMEABILITY					
GRAIN BOUNDARY CORROSION					
TEXTURE			YES	YES	
GRAIN SIZE					
GRAIN BOUNDARY SEGREGATION			YES	YES	
PHASE/CHEMICAL COMPOSITION			YES	YES	
LONGITUDINAL MODULUS					
SHEAR MODULUS					
BULK MODULUS					
YOUNG'S MODULUS		YES			
LAMÉ CONSTANT					
POISSON'S RATIO		BY CALCULATION			
DENSITY					
RADIOACTIVITY					

[illegible]

	YES						
	YES						
	YES [41,43,44]		YES [44,49]				YE [3]
	YES [41,44]		YES			YES	YE
	YES						
	YES					YES	YE
							YE [38,4]
							YE [38,4]
							YE [38,4]
	YES	YES	YES [44,49]			YES	YE [38,4]
							YE [38,4]
	YES	YES				YES	YE [38,4]
	YES [44,48]		YES [44,49]	YES [32]			YE [38,4]

	YES [39]				YES	YES
YES	YES	YES 42			YES	YES
						YES
YES	YES	MAYBE				MAYBE
	YES [38,43,44]					
	YES [38,43,44]					
	YES [38,43,44]					
YES	YES [38,43,44]					
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YES	YES [38,43,44]					
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