Determination of Residual Stress and Strain in Nb_3Sn Using the Nano-Indentation Technique

Shaun Murphy
National High Magnetic Field Laboratory
Florida State University, Tallahassee
Dr. Peter Kalu, Advisor
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Abstract

Residual stress and strain fields where investigated by use of the nano-indentation technique. Berkovich indentation tests where performed on samples machined to pre-determined specifications.

Introduction

Residual stresses and strains can be measured effectively using an indentation method. The most common type of indenters used are cone indenters, Vickers indenters, and Berkovich indenters. Figure 1 below shows a graphical representation of the indentation machine.

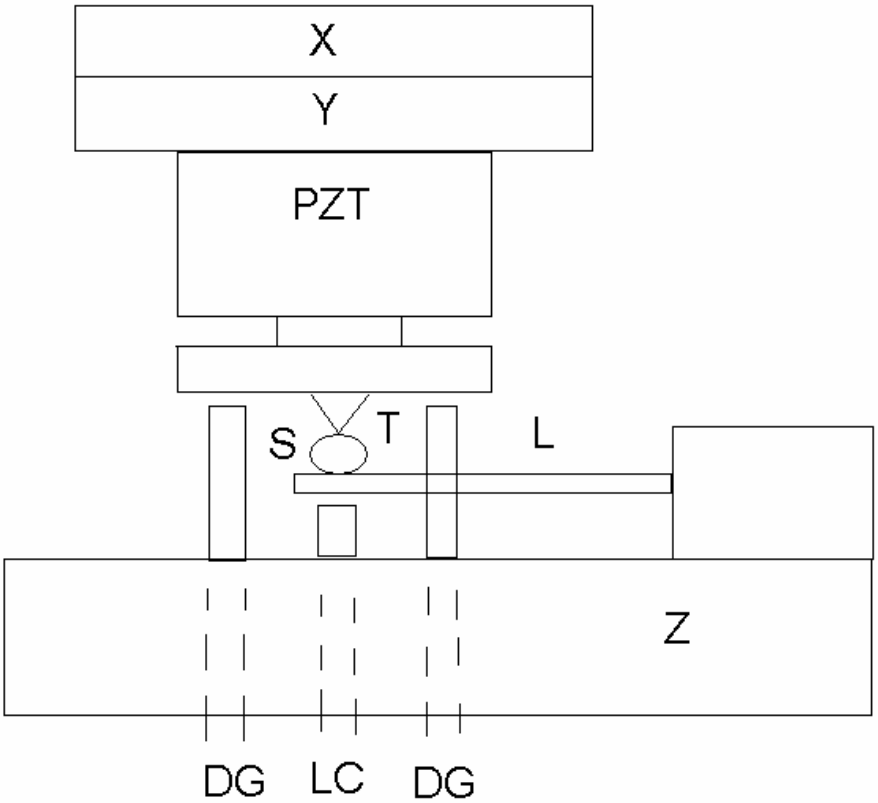

Figure 1 – Schematic of indentation machine.

The sample (S) is placed on a load arm (L), which the displacement as a function of load has been determined. The diamond indenter (T) is positioned in close range of the sample surface. The nano-indenter is moved by applying a small voltage to a piezoelectric transducer (PZT). The displacement of the sample can then be measured by the two capacitance gauges (DG). These two gauges along with the PZT allow the indenter to be moved at a constant rate. The deflection of the load arm can then be measured when the indenter makes contact with the sample by a third capacitance gauge (LC). Once the deflection of the load arm is known it can be converted to an applied load by using the load cell constant. The actual indentation depth can then be determined by subtracting the load arm displacement from the indenter displacement.[1,2] Normally either the indenter is pushed into a sample until the desired load or indentation depth is reached. The other parameter is then measured from the first. A load versus total indentation depth curve can then be easily obtained and represents both plastic and elastic deformation of the sample. The plastic indentation depth, x, can then be obtained by using Eqn. 1 below:

$$x = d - \frac{l}{s(l)} \qquad \text{Eqn. 1}$$

where d is the measured total indentation depth, l is the peak load, and s(l) is the unloading slope as a function of peak load. Also the hardness of the material can be found using Eqn. 2:[2]

$$H(x) = \frac{l(x)}{A(x)} \qquad \text{Eqn. 2}$$

where H(x) is the hardness, l(x) is the load at any plastic depth, and A(x) is the projected area at the same depth. This hardness is equivalent to the conventional hardness if the indenter does not have a large change of shape when the unloading process is executed.

Background

When performing nano-indentation to determine residual stresses in a material it is important to pay close attention to the contact area of the indenter. It has been found through previous experiments that the contact area should be determined using optical methods instead of by the nano-indentation machine. This is important due to the effects of piling in the sample, which cause the nano-indentation machine to inaccurately determine the contact area. In experiments run by Tsui et al.[3] and Bolshakov et al.[5] it was found that the piled up material, on the edges of the contact impressions, extend outward to a greater distance for the indentations stressed in compression, than for those stressed in tension. This leads to the assumption that the amount of pileup is greater for compression than tension. Further examination of the contact area led to more important results, the real, optically, measured contact area is independent of stress for the material used, aluminum alloy 8009. Also, the optically measured contact area is only in agreement with the nano-indentation machine when the largest tensile stressed samples were under consideration.

When examining the effects of pileup, it can be useful to quantify the results using an area ratio parameter. An area ratio parameter is the ratio of the true projected area of the contact including the pileup to the projected area that would be determined from the depth of penetration and the indenter geometry. This can be found using Eqn. 3 below:[5]

$$R = \frac{\pi r_c^{\,2}}{F(h_{\max})} \qquad \text{Eqn. 3}$$

where R is the area ratio parameter, r_c is the true radius of contact, $F(h_{max})$ is the empirically determined indenter shape function, and h_{max} is the maximum depth of penetration of the indenter. The lower limit on R, which occurs for the case of purely elastic contact, can be determined using Sneddon's analysis on the type of indenter used. Likewise an upper limit can be determined using Lockett's slip line field analysis, also for the type of indenter used. When using a rigid cone with a 70.3^o half included angle, it has been determined that pileup and sink-in can alter the contact area by as much as 60-70%.[5] The experiment referenced came to the conclusion that the hardness of aluminum alloy 8009 is not affected by applied or residual stresses, which is opposing most other reports on this topic. Also, it is possible that other methods used to determine the relation between hardness and applied or residual stresses have the same intrinsic measurement errors found in nano-indentation tests, causing the same errors as were found in the experiments run by Tsui et al.[3] and Bolshakov et al.[5] From experiments performed by Carlsson et al.[8] it can be concluded that the hardness value of a sample, with strain-hardening present, determined from nano-indentation testing, using optical methods of determining the contact ratio, is a function of residual strain only. If the sample in question lacks strain-hardening there is no mechanical test which can provide relevant information on residual strains.[6] The following equations are needed in order to perform a proper analysis to determine residual strains in a sample, which exhibits strain-hardening.

$$H = C\sigma(\varepsilon_{repr} + \varepsilon_{res}) \qquad \text{Eqn. 4}$$

$$H = K_1\sigma_1 + K_2\sigma_h \qquad \text{Eqn. 5}$$

$$H = K_1\sigma(.02 + \varepsilon_{res}) + K_2\sigma(.35 + \varepsilon_{res}) \qquad \text{Eqn. 6}$$

where H is the hardness of the material, ε_{res} is the residual strain, and C, K_1, and K_2 are constants determined by the type of indenter used, $\sigma(\varepsilon_{repr})$ is flow stress at a representative value of the plastic strain, and σ_1 and σ_2 are given as $\sigma(.02)$ and $\sigma(.35)$, respectively. Eqn. 6 is a variation of Eqn. 5 which gives a better representation of the how the hardness of a material is affected by residual strains. Eqn. 6 should be used for irregularly hardening materials which do not satisfy Eqn. 4. In order to properly determine residual strain fields by indentation, first the constants in one of the above equations should be determined using a virgin sample of the material in question, as long as the strain hardening of the material is known. The residual strain field can then be determined by comparing the hardness found to the hardness in a situation with residual strains present.[6]

In order to fully understand the effects of residual strains on a sample two more parameters must be introduced.

$$\Lambda = \frac{E \cdot \tan(\beta)}{\sigma_Y (1-\nu^2)} \qquad \text{Eqn. 7}$$

$$c^2 = \frac{A}{A_{nom}} \qquad \text{Eqn. 8}$$

$$c^2 = C_1 + C_2 \ln(\Lambda) \qquad \text{Eqn. 9}$$

where Λ is a parameter used to characterize the level for which the sample falls into as shown in Figure 2, E is Young's modulus, ν is Piosson's ratio, σ_Y is the flow stress, β is the angle between the sharp indenter and the undeformed surface of the material, c^2 is an indentation parameter known as the area ratio, A_{nom} is the nominal contact area as defined by the type of indenter used, and C_1 and C_2 are constants. It is important to note that Eqn. 9 is valid for approximately $5<\Lambda<400$. If $c^2<1$ sinking-in occurs whereas if $c^2>1$ than piling-up is present.[6]

Residual stresses, according to experiments by Carlsson et al.[6] have no effect on the hardness of a material; however, they do have an effect on the area ratio c^2. It was found that a tensile residual stress will give a smaller value of the area ratio, compared to indentation of a stress-free material; whereas, a compressive test increases the c^2value. This could allow for the determination of residual stresses by comparing the c^2 values of a virgin material to one with residual stresses. More accurate results can be obtained by modifying Eqn.'s 7 and 8 as shown below.

$$\Lambda = \frac{E \cdot \tan(\beta)}{(\sigma_Y + \sigma_{res})(1-\nu^2)} \qquad \text{Eqn. 10}$$

$$c^2 = C_1 + C_2 \ln\left(\frac{E \cdot \tan(\beta)}{\sigma_Y (1-\nu^2)}\right) + C_2 \ln\left(1 + \frac{\sigma_{res}}{\sigma_Y}\right) \qquad \text{Eqn. 11}$$

where C_2 can be determined experimentally and was found to a value of .32. Eqn. 11 can be further simplified by realizing the first two terms are simply the area ratio for the virgin material as shown below.[6]

$$c^2 = c^2(\sigma_{res} = 0) - .32 \ln\left(1 + \frac{\sigma_{res}}{\sigma_Y}\right) \qquad \text{Eqn. 12}$$

It is important to note that this is only an approximate relation and is also only valid for Λ between 5 and 400. For a strain-hardened material it has been found that Eqn. 12 is also valid.

Experimental Procedure

The indenter used in this experiment was a Berkovich indenter mounted on a nano-indentation testing device. In this experiment the indentation load P was set to 30 mN. The load versus displacement of the indenter head h was recorded during the loading and unloading processes. The rate of both the loading and unloading was approximately 60 mN/min. A pause of 10 seconds was used at the end of the loading portion of the experiment.

The true area of the indentation was not deduced from the nano-indentation data but was found with the help of three-dimensional pictures taen of the indentation using an atomic force microscope (AFM). However, in order to find the area coefficient presented in Eqn. 8 the area, A_{nom}, from the indentation machine was used. From the known values of P, h, and A_{nom} the hardness and area ratio were determined. One object of note is that the actual process of indenting a material can introduce residual stresses and strains into the material. Placing the indentation an appropriate distance, in this case 30 μm, apart, counteracted this. Both samples of Nb_3Sn and virgin copper were examined in order to better understand the results that were obtained. Also two separate sections of Nb_3Sn were examined, the copper stabilizer and copper tin sections of the sample.

Conclusions

All of the relevant data for the experiment performed is shown in Table 1 below.

Table 1 – Relevant Data

	Hardness (MPa)	c^2	σ_{res} (MPa)
Cu (pure)	1560	1.005	N/A
Cu Stab (cross)	1332	1.030	-4.51
Cu Stab (axial)	1355	1.058	13.4
Cu/Sn (cross)	2244	0.941	N/A
Cu/Sn (axial)	2123	0.987	N/A

As can be seen in the above table the hardness for both the axial and cross sections of the copper stabilizer are lower than the hardness of the virgin copper. This leads to the assumption that there are no residual strains present in the copper stabilizer because residual strain is a function of hardness where if the hardness of a material is greater than that of an un-processed sample of the same material than residual strains are present. However, if the hardness is smaller than residual strains are not present. When examining the copper tin it is impossible to say whether there are or are not residual strains present because no un-processed sample of the copper tin found in Nb_3Sn was available for testing. The increases in hardness is due, at least in part, to the solid solution relation that tin has on copper in the formation of copper tin. Also it is impossible to determine the residual stresses in the copper tin section of Nb_3Sn for the same reasons as stated above. However the residual stresses present in the copper stabilizer where calculated and are presented in the table above. The most important conclusions can be summarized as follows:

- Residual Stresses are present in the copper stabilizer but are of a very small value
- There are no residual strains present in the sample
- The residual stresses in the axial and cross sections of the copper tin are of similar value; however, there exact value cannot be determined from this experiment
- The Nano-indentation technique is a viable option for determining residual stresses
- The subject requires further additional study

References

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