Vacancy Concentration in Nickel through Nanoindentation

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BACKGROUND

Nanoindentation is the most common way to test small volumes of materials for mechanical properties. This process consists of a sharp diamond tip indenting the surface of the desired material. Once the tip is removed it leaves an indent from which hardness is calculated from, through other software modulus, roughness, and other mechanical properties can be determined.

In the Positron Analysis, a beam of positrons are injected into the sample. When annihilation occurs the vacancy concentration can be determined for the sample.

If once sample was to contain a higher vacancy concentration it will appear in the spectra. The effect of vacancy concentration in yield point data is investigated in this project by the following steps.

PROCEDURE

Preparation of Nickel Samples
Grinding, Polishing, Electro-polishing
First round of automations
Data collection, Roughness values and Multiple curve analysis
Positron Analysis
Vacancy concentration
Calculations
Average and instantaneous tip radii
Calculating maximum pressure (p0), maximum shear stress (tmax), and area
Encapsulation in vacuum
Heat treatment
Second round of automation
Data collection, New roughness values and MC values
Calculations
Calculating new average and instantaneous tip radii
Calculating p0, tmax, area

RESULTS

In this research project, the objective was to determine the effect that vacancy concentration has on the yield behavior in nickel. By calculating the maximum shear stress (tmax) through an average radii and instantaneous radii it shows the difference between the two tmax values. When calculating tmax through the first method the elastic deformation section of yield point graph was fitted to the Hertz equation, this result in an average radius. When assuming instantaneous radii the load and displacement at the yield point of each indent was used in the second tmax formula. When tmax is plotted vs. probability it can be observed that at low yield loads the two tmax values are very close to each other; however, at higher loads the instantaneous radii curve starts deviating to higher tmax values while the average radii curve maintains a steeper slope. It can be determined that after heat treatment the probability with respect to tmax increases as shown in figure 1 and 3. In both figures a graph of tmax vs. f-probability is displayed with two curves (before and after heat treatment). With either tip, tmax increases considerably after heat treatment.

It appears that by changing the vacancy concentration it will also change the yield behavior and subsequently tmax. The nickel sample was heat treated at 1023°C and slowly cooled. In fig 6, it can be determined that before heat treatment the vacancy concentration was around 1.15 at 2-3 μM. After heat treatment the vacancy concentration was lowered to nearly 1.0 at the same depth, which is considered as nearly vacancy free.

CONCLUSION

By altering a nickel sample through heat treatment the yield behavior was determined and compared to the yield behavior before heat treatment. The results concluded that after heat treatment the maximum shear stress increases considerably compared to the sample before heat treatment. This complies with the results from positron data, before the heat treatment the vacancy concentration was much higher than after the alteration. The slow cool after heat treatment resulted in annealing vacancies out of the sample and resulting in higher tmax values. Thus, heat treatment with a slow quench will produce a higher tmax value for the yield behavior.

Formulas
Hertz equation
- tmax = (Average radii)
- tmax = (Instantaneous radii)
- Area under tip

Figures
Fig. 1: Yield point probability graph with the smaller tip before and after HT.
Fig. 2: A graph of a high load yield point after HT. Fig. 2.b Graph of a low load yield point after HT. Fig. 3: Yield point probability graph with the larger tip before and after HT.
Fig. 4: Curve fitting on elastic part of a yield point graph, calculates the tip radius for the larger tip after HT. Fig. 5: Shows a tmax vs. f-probability graph for the smaller tip with average and instantaneous radius tip.

Fig. 6: Positron Analysis shows that sample NIS 5 (#5) before heat treatment has a higher vacancy concentration then after heat treatment with a low quench, suggesting the low quench annealed vacancies out of the sample.

Fig. 7: Roughness image of nickel sample; note the height scale is extremely exaggerated.

References
Nanoinentation. “Introduction to Nanoindentation.”
www.Nanoindentation.cornell.edu
“Positron Analysis.” www.uwo.ca/isw/facilities/positron/index.html

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