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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 (τmax), 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, τmax, area

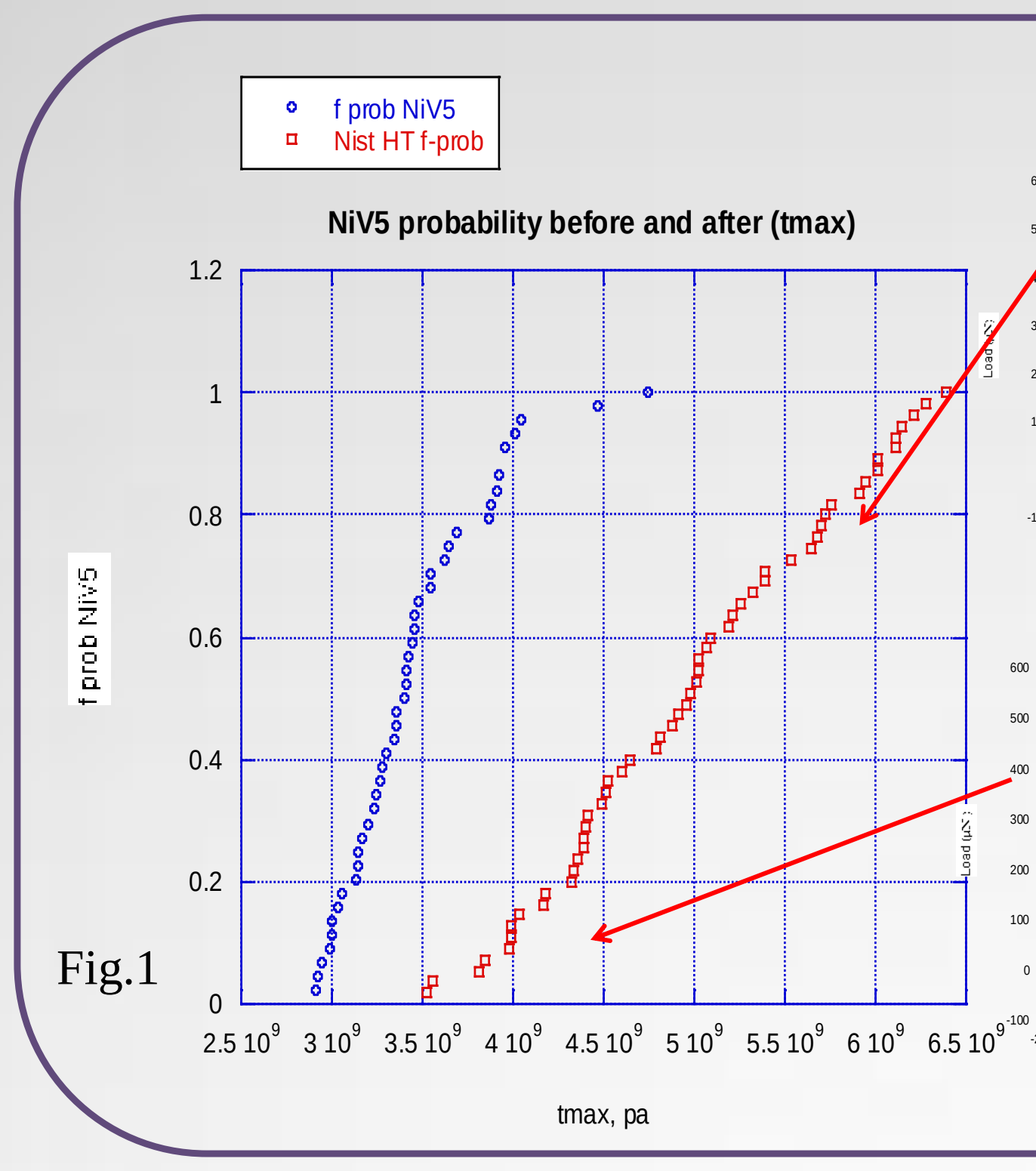
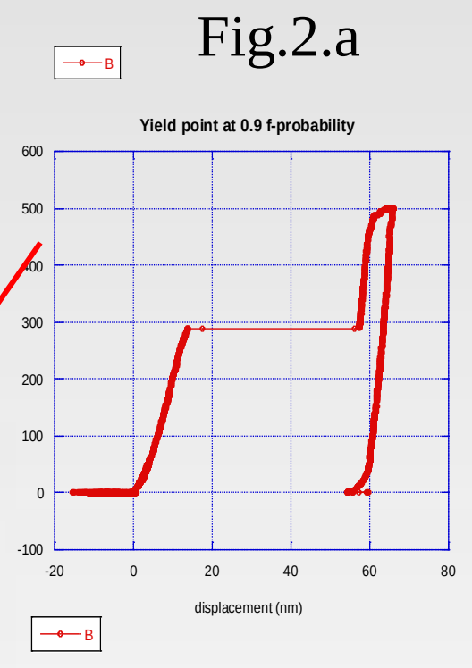


Fig.1

Fig.2.a



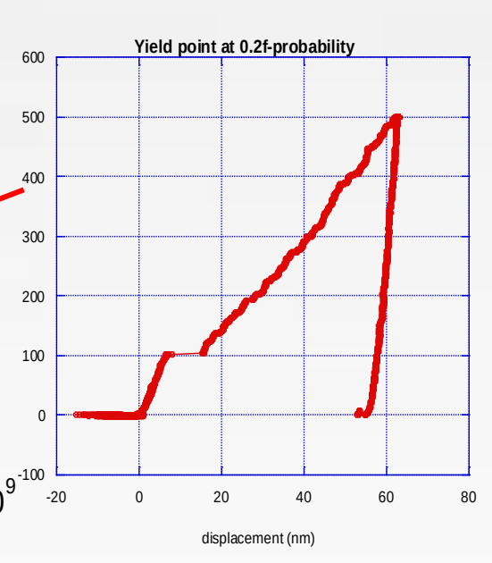


Fig.2.b

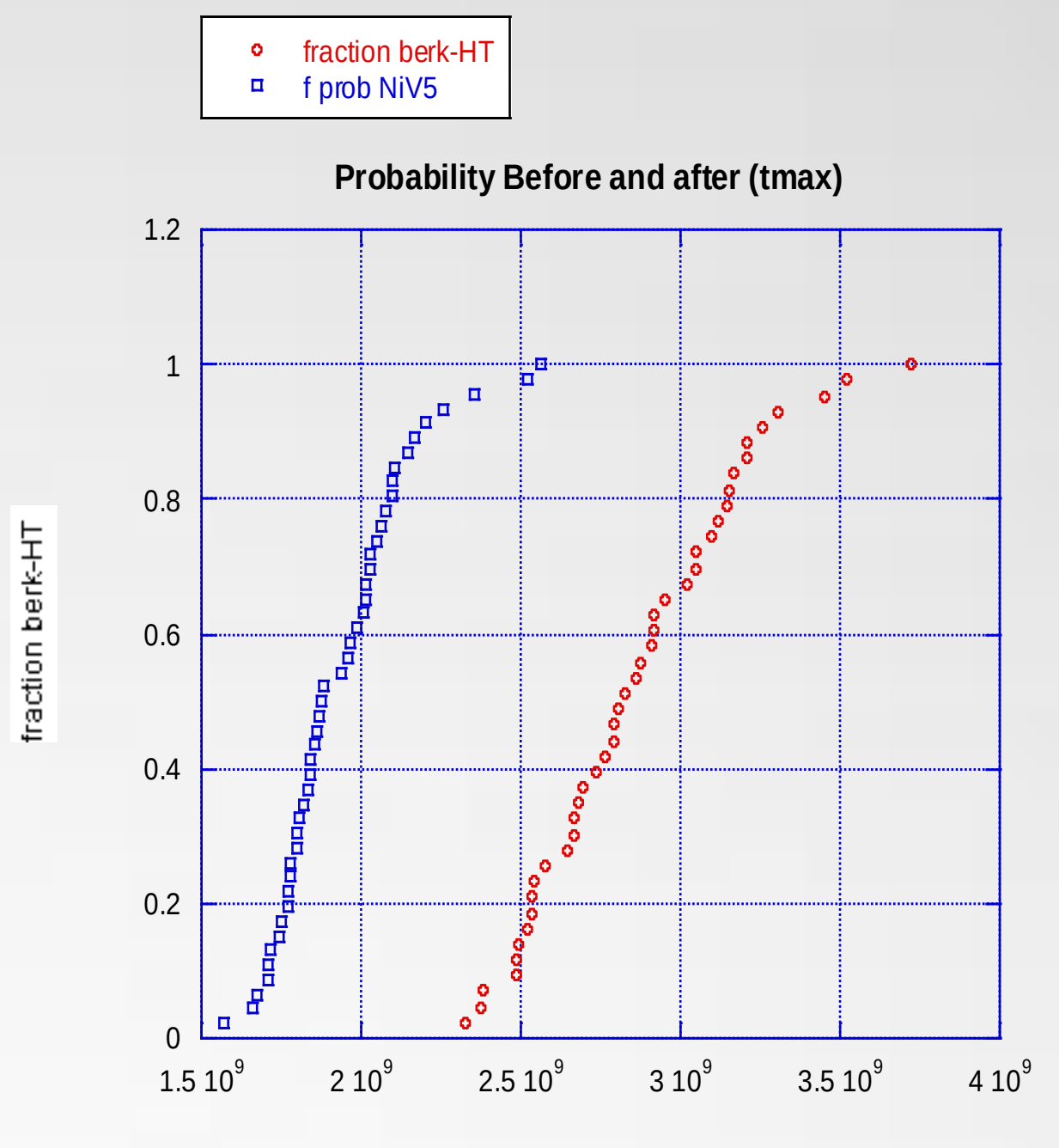
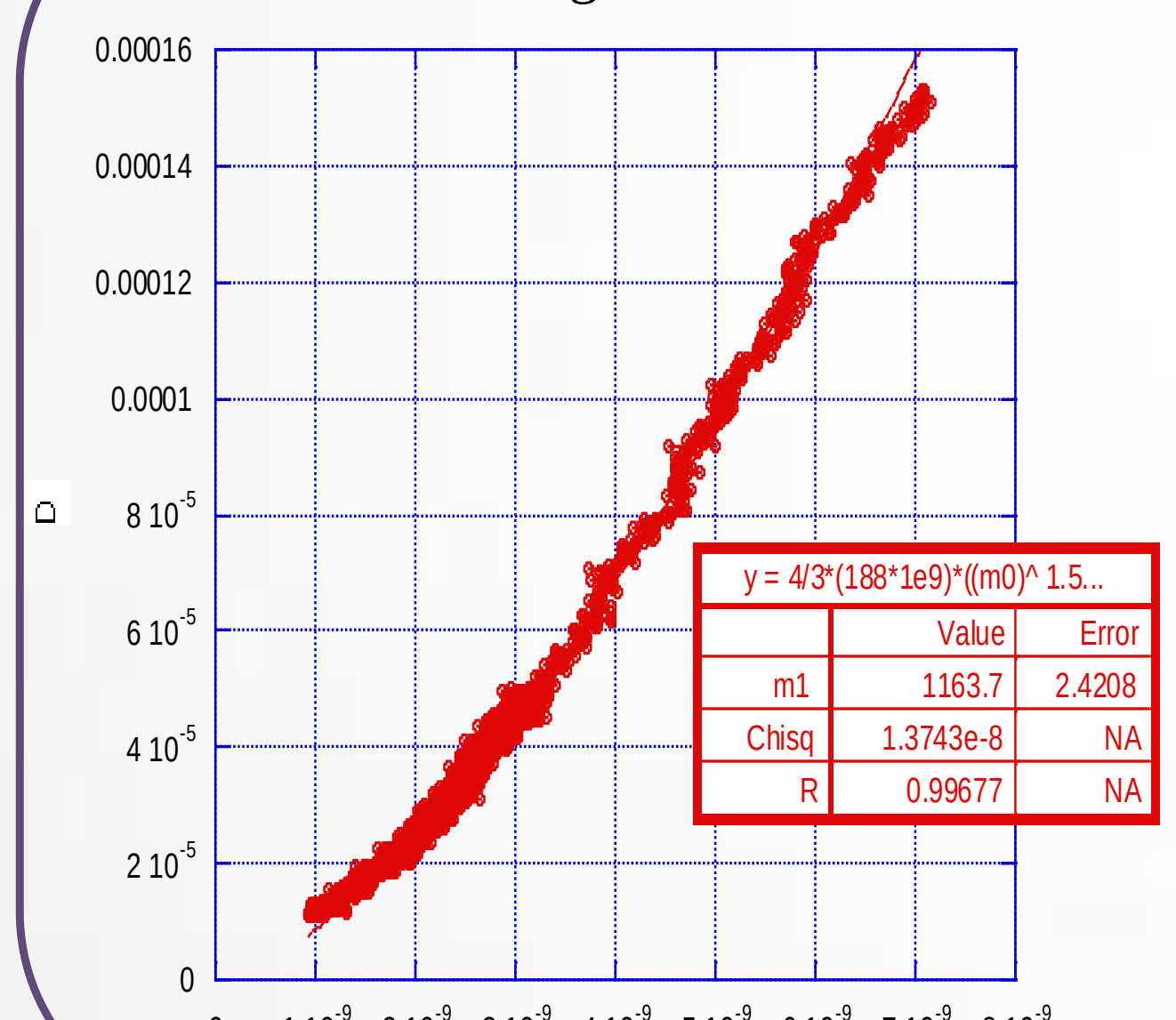


Fig.3

Fig.1: yield point probability graph with the smaller tip before and after HT.
Fig2.A: graph of a high load yield point after HT. **Fig2.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



y = 4/3*(188*1e9)*((m0)^ 1.5...		
	Value	Error
m1	1163.7	2.4208
Chisq	1.3743e-8	NA
R	0.99677	NA

Fig.5

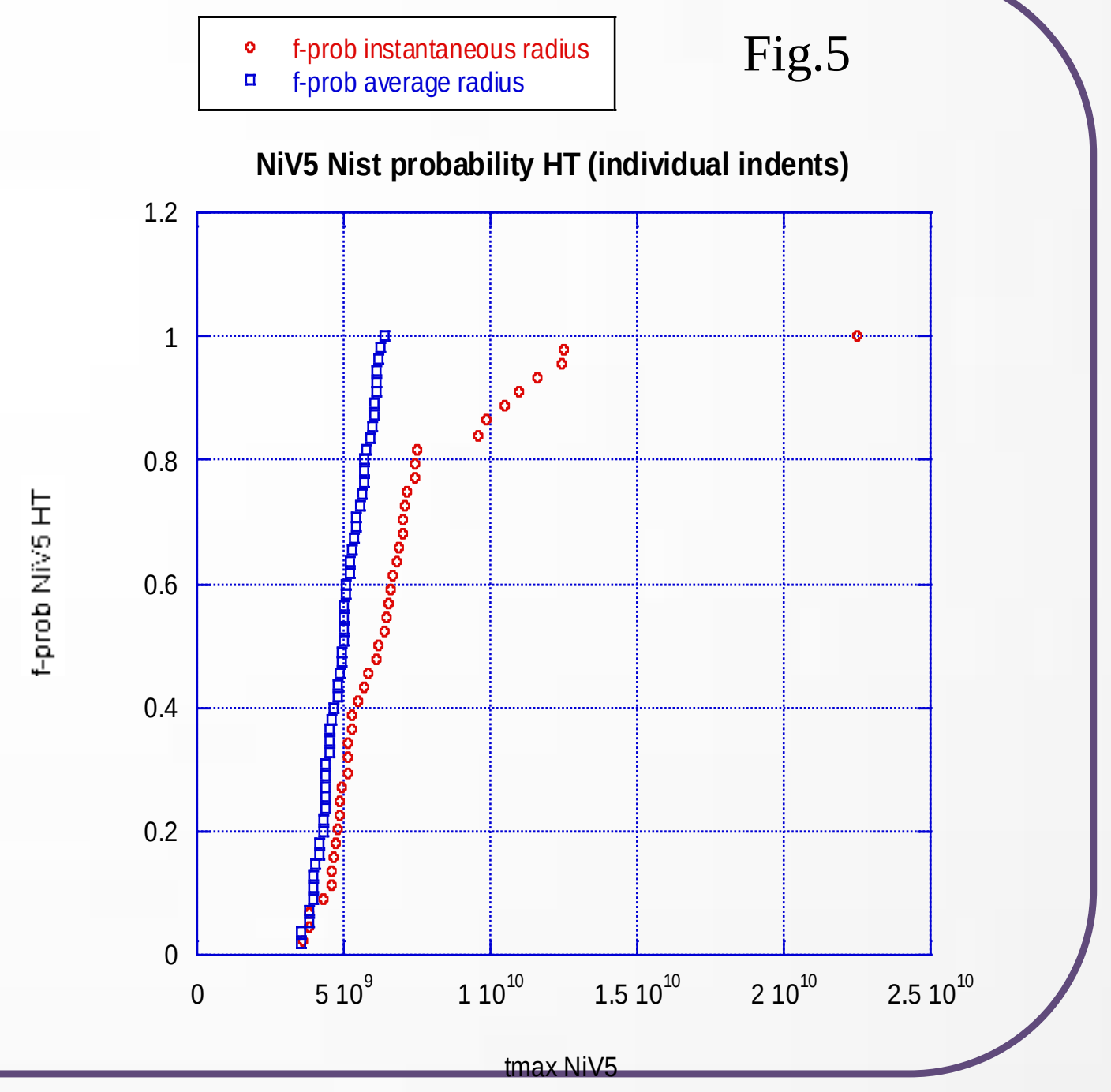


Fig4. Curve fitting on elastic part of a yield point graph, calculates the tip radius for the larger tip after HT. **Fig5.** Shows a τmax vs. f-probability graph for the smaller tip with average and instantaneous radius tip.

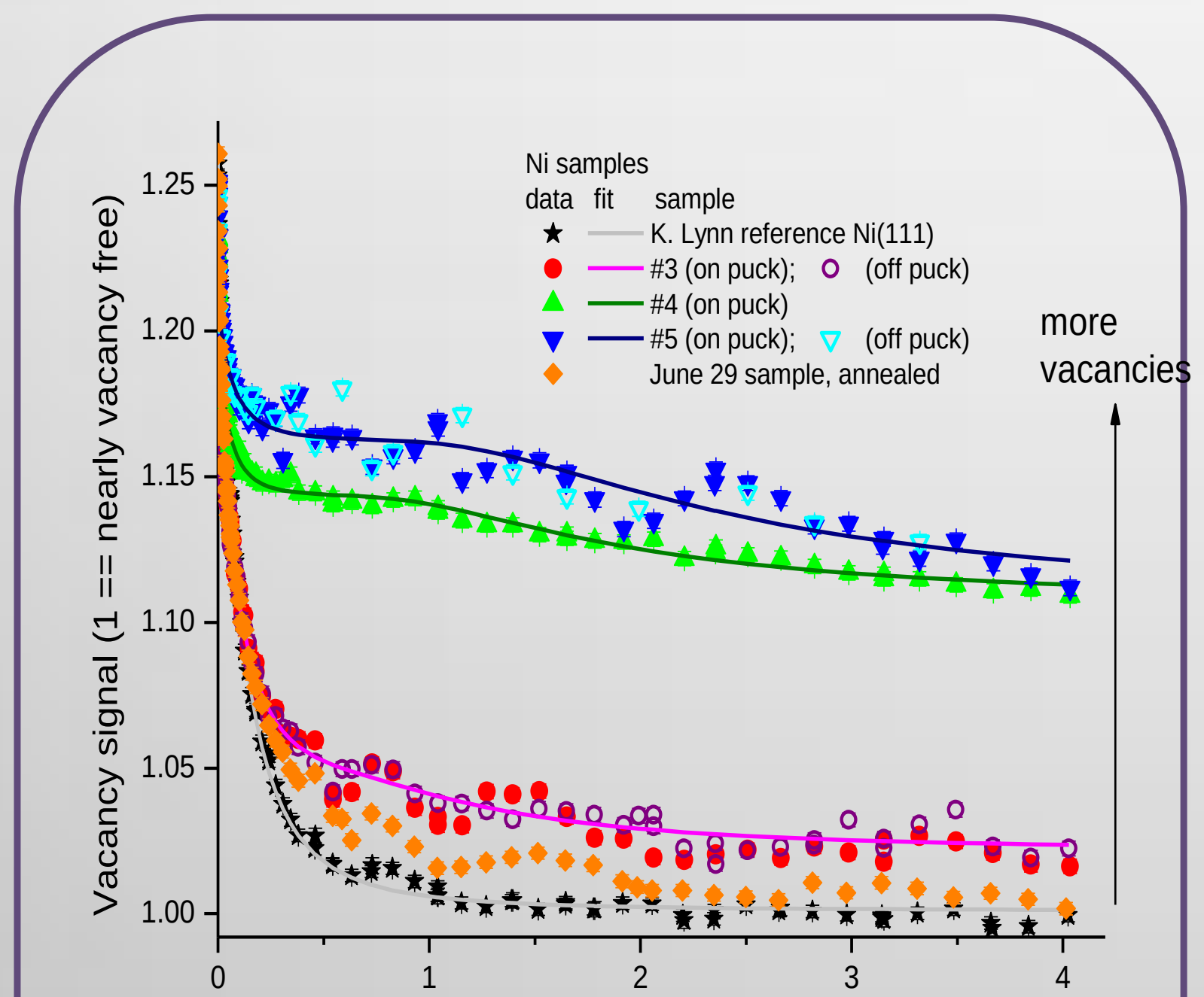


Fig. 6

Fig. 6: Positron Analysis shows that sample NiV5 (#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.

Formulas

$\frac{4}{3}(188 * 10^9)((m0)^{3/2})(m1 * 10^{-9})^{\frac{1}{2}}; m1 = 3$ Hertz equation

$pm = \left(\frac{6E^{*2}}{\pi^3 R^2}\right)^{1/3} p^{1/3}$ Maximum pressure

$tmax = 0.31pm$ τmax (average radii)

$Tmax = 0.31\left(\frac{8}{3\pi}\right)\left(\frac{E^{*2}\int^{2}}{p}\right)$ τmax (instantaneous)

$a = \left(\frac{3RP}{4E^*}\right)^{1/3}$ $c2 = c1/cmax(c1)$ Area under tip

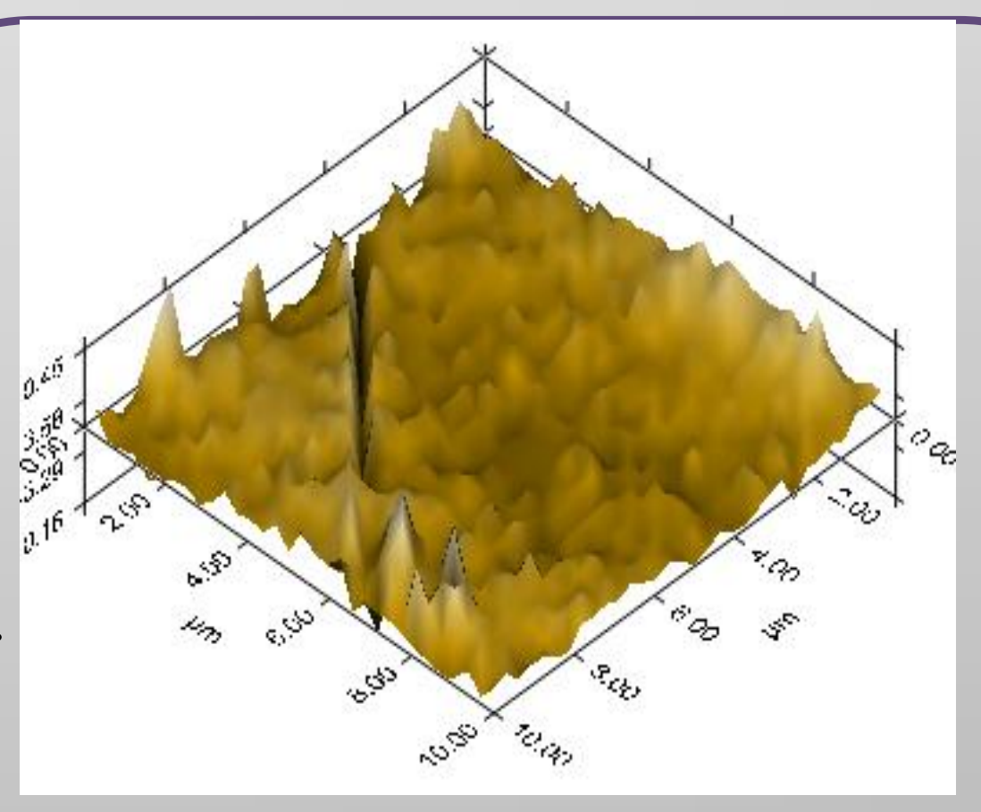


Fig. 7

Fig7. Roughness image of nickel sample; note the height scale is extremely exaggerated.

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 sheer stress (τmax) through an average radii and instantaneous radii it shows the difference between the two τmax values. When calculating τmax 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 τmax formula. When τmax is plotted vs. probability it can be observed that at low yield loads the two τmax values are very close to each other; however, at higher loads the instantaneous radii curve starts deviating to higher τmax values while the average radii curve maintains a steeper slope (fig.5).

It can be determined that after heat treatment the probability with respect to τmax increases as shown in fig 1 and 3. In both figures a graph of τmax vs. f-probability is displayed with two curves (before and after heat treatment). With either tip, τmax increases considerably after heat treatment.

It appears that by changing the vacancy concentration it will also change the yield behavior and subsequently τmax. 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 sheer 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 τmax values. Thus, heat treatment with a slow quench will produce a higher τmax value for the yield behavior.

References

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