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# SUPERPLASTICITY AND MICROSTRUCTURE EVOLUTION IN NANONICKEL

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**Abstract.** Although superplasticity has been studied for decades, few observations of this behavior have been reported for pure metals. A problem generally attributed to the difficulty of maintaining a grain size small enough for superplastic flow at the temperatures required for superplasticity. With the produce of nanocrystalline (NC) materials it has been hoped that superplasticity could be obtained in a number of pure metals due to the large volume fraction of grain boundaries in nanocrystalline material should enhance the tendency for superplastic behavior. Two methods of nanocrystalline materials producing (severe plastic deformation and elctrodeposition) allow making fully dense samples available for testing. In this paper, superplasticity was obtained from electrodeposited nickel with initially nanocrystalline structure at the lowest normalized superplastic temperature reported for any crystalline material. A maximum elongation of 895% was obtained at $0.4 \cdot T_M$. Although a mean grain size of ultrafine-grained (UFG) microstructure obtained by severe plastic deformation is comparable with grain size of NC nickel at the beginning of tensile test (about 0.5 μm), no superplastic behavior of UFG samples was detected. This controversy is discussed from viewpoint of non-homogeneity of microstructure in UFG pure nickel.

## 1. INTRODUCTION

A strong refinement of microstructure in metals and alloys by high-pressure torsion (HPT) furnishes a potential to obtain new and extraordinary properties [1–3], which are grain size dependent. Bulk nanocrystalline (NC) and ultrafine-grained (UFG) materials provide a good opportunity to study a scalability of physical and mechanical properties to nanoscale range. Superplasticity is known as strong grain size dependent phenomenon. It is manifested as an ability of a material to sustain large plastic deformation. Necessary conditions for superplasticity observation are a stable fine-grained microstructure and a temperature higher a half of the melting temperature. The generalized constitutive equation for superplasticity links the flow stress and strain rate:

$$\dot{\varepsilon} = A \frac{DGb}{kT} \left( \frac{b}{d} \right)^p \left( \frac{\sigma}{G} \right)^n, \qquad (1)$$

where $\dot{\varepsilon}$ is the strain rate, $D$ is the appropriate diffusivity (lattice or grain boundary), $G$ is the shear modulus, $b$ is the Burger's vector, $k$ is the Boltzmann's constant, $T$ is the test temperature, $d$ is the grain size, $p$ is the grain size exponent and $\sigma$ is the applied stress and $n$ is resiprocal to the strain rate sensitivity. This equation suggests that NC and UFG materials, at constant strain rate, may be superplastic at significantly lower temperatures than their coarse-grained counterparts, which would reduce the problem of grain growth during deformation.

In this study, superplasticity was obtained from electrodeposited nanocrystalline nickel at 350 °C, which is 0.36 of the melting point ($T_M$), the lowest normalized superplastic temperature reported for any crystalline material. A maximum elongation of 895% was obtained at $0.4 \cdot T_M$, which is a large reduction in temperature and increase in elongation over the previously reported best result for nickel [4] of 250% elongation at $0.75 \cdot T_M$. This result is discussed from viewpoint of unsuccessful attempts to attain superplasticity in UFG pure metals processed by high-pressure torsion. Although in many UFG alloys, superplastic behavior at substantially low temperature and at high strain rate was found [5, 6] pure copper and nickel refined by HTP have not demonstrated noteworthy plasticity [7]. This controversy is discussed from viewpoint of non-homogeneity of microstructure observed in UFG pure nickel.

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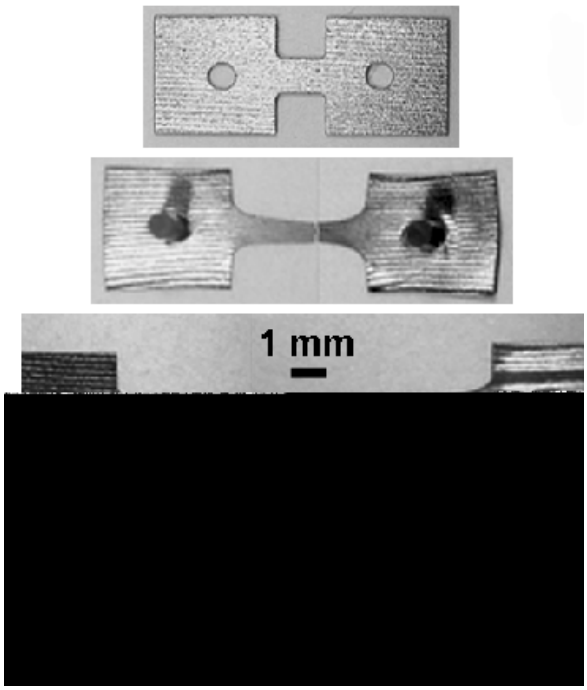


**Fig. 1.** Tensile specimens in the as-machined geometry, and deformed at a strain rate of $10^{-3}$ $s^{-1}$. Deformation temperatures and final elongation are indicated.

## 2. EXPERIMENT AND RESULTS

Tensile specimens having a gage length and width of 1.0 mm and a nominal thickness of 0.2 mm were electro-discharge machined from nickel. Testing was conducted at constant strain rates using a laboratory constructed bench-top tensile machine having 5 μm displacement resolution and 10 to 20 g load resolution. Temperature was controlled to ± 1 °C. Final elongation was measured in the gage length eliminate contribution from deformation in the specimen heads. Microstructure characterization of the material under study was performed by microhardness measurements, transmission electron microscopy (TEM) and X-ray diffraction. Crystallite size in NC nickel was characterized using both Scherrer and Warren-Averbach methods. Differential scanning calorimetry (DSC) was performed on the as-received material.

**Nanocrystalline nickel.** The as-received microstructure has the small initial grain size of 35 nm from dark field TEM that is in general agreement with the values given by x-ray diffraction. Tensile samples before and after deformation are shown in Fig. 1, which demonstrates the large uniform elongation and absence of localized necking representative of superplasticity. Stress-strain curves obtained at a constant strain rate of $10^{-3}$ $s^{-1}$ are shown in Fig. 2. An important transition in mechanical behavior occurred above 280 °C, indicated by the 350 °C curve that shows yielding and strong strain hardening with a large increase of deformation into the superplastic regime of greater than 200% elongation. This transition from low plasticity to large plasticity coincided with an exothermic peak in the DSC curve as shown in Fig. 3, where elongation as a function of test temperature is combined with a plot the DSC curve. The exothermic peak has been shown by Wang et al [8] to be associated with grain growth in nanocrystalline nickel. A maximum elongation of 895% was obtained at 420º C and strain rate of $10^{-3}$ $s^{-1}$. An elongation of 295% was obtained at 350 °C and a strain rate of $10^{-3}$ $s^{-1}$. Based on the heat-

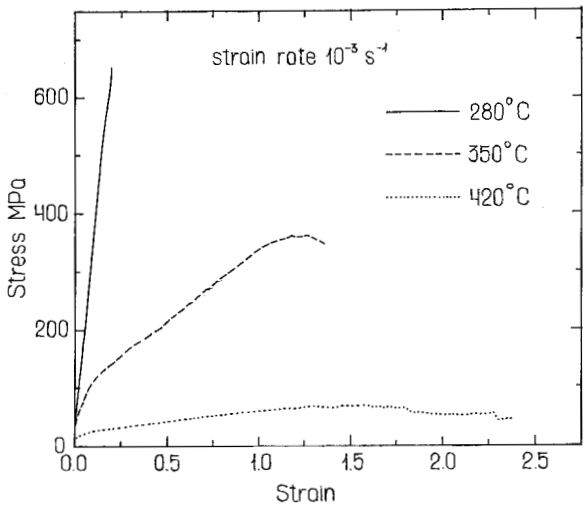


**Fig. 2.** Stress-strain curves show a transition in mechanical behaviour from low plasticity to superplasticity above 280 °C.

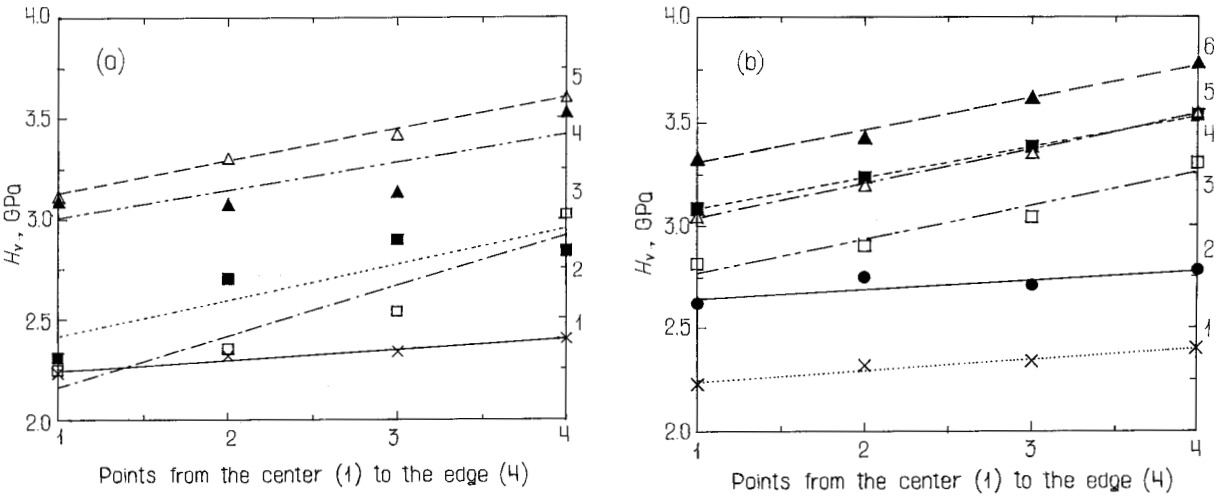


**Fig. 5.** Microhardness vs. distance across the HPT disk as a function of applied pressure (a) and number of whole rotation (b). Microhardness of compressed disk (no rotations) is shown for comparison.

grain growth due to the very large volume fraction of grain boundaries, which increases the internal energy of the system. Because grain boundary migration has similar activation energy as for the mechanisms of superplasticity, if sufficient thermal energy is available for superplastic flow, grain growth will occur. This competition between grain growth and superplasticity was established early in the development of the field, but has not been investigated at such small starting grain size and low homologous temperatures. In nanocrystalline nickel, rapid grain growth during superplasticity, even at low homologous temperatures, has the effect that only a small part of the deformation can occur in the nanocrystalline state. It appears as though the driving force for grain growth is sufficiently reduced by a grain size increase to approximately 1 μm, that superplastic mechanisms can operate competitively with grain growth in the temperature range from around

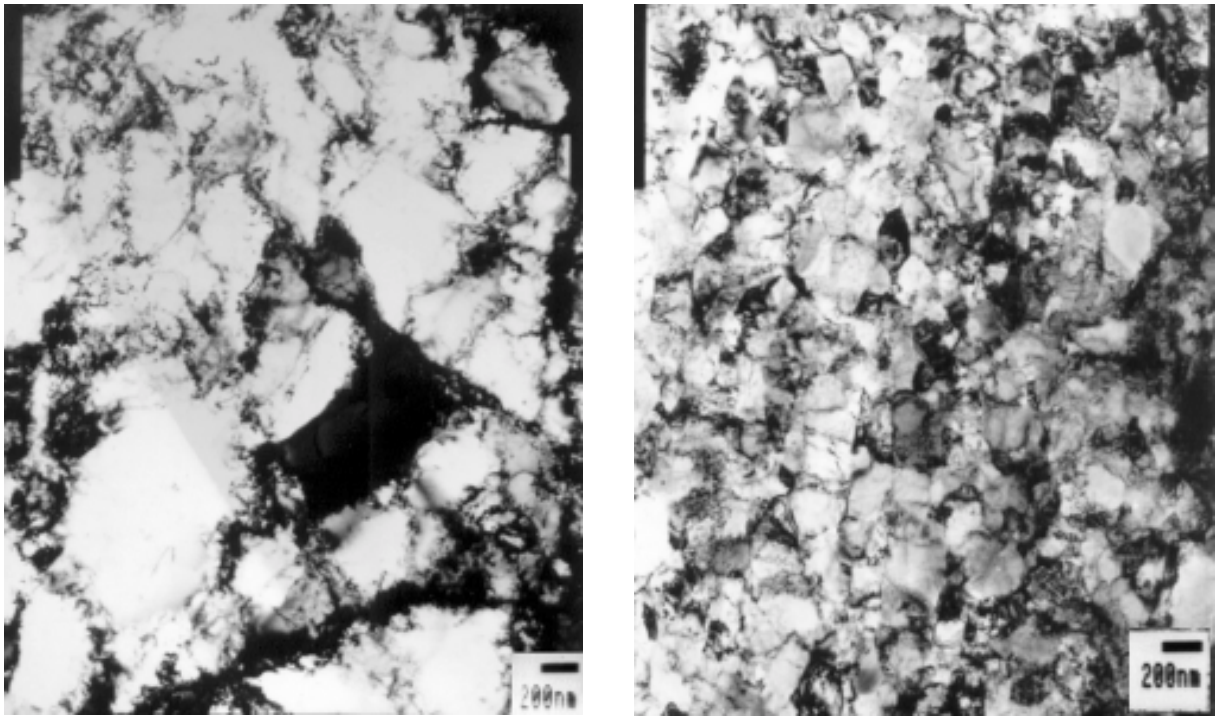


**Fig. 6.** Typical microstructure of the nickel deformed at 1 GPa pressure (5 turns) taken from the centre (a) and from the edge (b) of the sample.

350 °C to around 420 °C, since the final grain size at both of these temperatures was in the 1.0 μm range.

**Ultrafine-grained nickel.** The present results show a linear dependence in microhardness along the diameter of the disk. It consists of the equation used for the strain value calculation: $\gamma = (\theta \cdot r) / h$ with non-zero value of strain in the center of the sample. One can see saturation in gain of microhardness with increase of applied pressure and of stored strain (number of whole rotations), but the inclination of plots doesn't change. This means keeping differences in microstructures of the middle and the edge of disks. TEM investigation shows considerable distinctions in the mean grain size. The mean grain size is about 1 μm in the microstructure taken from the center and 0.1 – 0.2 μm for the foil cut from the edge. Non-homogeneity of the samples after HPT might be reasonable explanation for unsuccessful attempts to obtain superplastic behaviors of UFG pure metals.

## 4. CONCLUDING REMARKS

Superplasticity has been obtained from nanocrystalline electrodeposited pure nickel in the temperature range of 350 ºC to 490 ºC. An elongation of 295% was achieved at 350 °C ($0.36T_M$). This is the lowest normalized superplastic temperature reported for crystalline materials. A maximum elongation of 895%, measured in the gage section, was obtained at a strain rate of $10^{-3}$ $s^{-1}$ and a temperature of 420 ºC.

Ultrafine-grained nickel processed by HPT has demonstrated residual non-homogeneity in the samples. It decreases with increase of applied pressure and number of whole rotations but still can be detected by microhardness measurement. The non-homogeneous microstructure across the UFG nickel disk may shed light on what is a cause of unsuccessful attempts to find superplasticity in UFG nickel after high-pressure torsion.

## ACKNOWLEDGMENTS

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