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Microstructural evolution and strength variability in microwires

S. Agepati [1], P. Ghosh [*,2], A.H. Chokshi

Department of Materials Engineering, Indian Institute of Science, Bangalore 560012, India

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ABSTRACT

Tensile experiments on cold-drawn Ni microwires with diameters from ~115 to 50 μm revealed high strengths, with significant strength variability for finer wires with diameters less than ~50 μm. The wires showed pronounced necking at fracture. The coarser wires with diameters >50 μm exhibited conventional ductile cup-cone fracture, with dimples in the central zone and peripheral shear lips, whereas finer wires failed by shear with knife or chisel-edge fractures. Shear bands were observed in all samples. Further, through- section microscopy of selected fractured samples revealed that the shear bands did not go across the enitre specimen for the coarser wires. The shear bands led to grain fragmention, with a reduction in grain aspect ratio as well as rotations away from the initial <111> orientations. The strength data were analysed based on a Weibull approach. The data could be rationalized in terms of failure from volume defects in coarser wires, with a high Weibull modulus, and from surface defects in finer wires, with a low Weibull modulus and greater variability.

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1. Introduction

The strength of ductile metals is governed by factors that influence dislocation mobility, such as dislocation spacing or grain size (d), and the strength is usually not dependent on external specimen dimensions of engineering components [1]. For brittle ceramics, strength is controlled primarily by the distribution in the population of flaws such as cracks or pores. In this case, the strength typically increases with a decrease in its volume (related to lower probability of finding a large critical flaw), giving rise to an external size effect on strength [2].

Early tensile studies on metallic whiskers in the 1950s and 1960s, displaying very high strengths approaching the theoretical values, revealed an external size effect in specimens with a low initial dislocation density and typical diameters of $\Phi < 10\ \mu m$ [3,4]. Inspection of the available data suggests that there was considerable variation in strengths of the whiskers at smaller diameters compared to coarser whiskers.

External size effects have been reported recently in the compression deformation of micro and nano-pillars made from single crystalline metals and alloys, where the yield strength increases with a decrease in the pillar diameter, which have been discussed in terms of the need to nucleate dislocation or to the stochastic operation of weak dislocation sources in small specimens [5–7]. External size effects have also been noted in conventional large metal specimens tested under a strain gradient [8,9], as in torsion, bending or indentation, with an additional contribution to the total dislocation density by geometrically necessary dislocations (GND). In polycrystals, the need for compatible deformation introduces a GND contribution to strengthening, even under uniaxial deformation. However, since the surface grains in uniaxial deformation experience lower constraints compared to grains in the sample interior, it is anticipated that the strength will decrease with a reduction in the sample size when there are only a few grains across a section [10]. A recent study has demonstrated that both weakening and strengthening can occur in polycrystals in a complex manner that cannot be accounted for by a simple term such as the ratio of the sample diameter to the grain size [11].

In several structural materials the desired intrinsic length scales are engineered by the manufacturing process. For instance, the high strength pearlitic steel wires used in suspension bridge cables and the copper–niobium or copper–silver wires used for long pulse high field resistive magnets are processed by wire drawing the as-cast ingots or wire bundles to a high true strain of ~10 [12,13]. During the wire drawing process the initial random eutectic lamellar or dendritic structure is re-oriented and subsequently refined to a scale of few tens of nanometers, providing strengths of 2–6 GPa in the steel wires [14,15]. The limited dislocation activity at such fine length scales often introduces localized shear bands as a dominant deformation process [16,17]. Thus, it is important to understand the influence of external size on

* Corresponding author.
E-mail address: Pradipta.ghosh@oeaw.ac.at (P. Ghosh).
[1] Now at Applied Materials India PVT LTD, India.
[2] Now at Erich Schmid Institute of Material Science, Leoben, Austria.

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shear banding mechanisms in conventionally drawn metallic wires.

Warthi et al. [18] noted recently, from limited experiments, an increase in scatter of fracture strength with decreasing wire diameter Φ, and observed two different size dependent failure modes in commercially drawn polycrystalline nickel (Ni) wires together with shear banding. For coarser wires ($\Phi > 50$ μm) void nucleation and growth in the central necked region and shear lips at the periphery led to dimpled fracture surfaces. However, finer ($\Phi < 50$ μm) wires showed limited cavitation and a chisel shaped failure mode.

A common factor in many studies with fine structures, be they whiskers, micropillars or wires, is the tendency towards increasing scatter in data at smaller dimensions. However, analysis of such scatter is limited, especially over size ranges where there is a difference in fracture modes. The present study was undertaken for understanding the deformation processes in polycrystalline nickel microwires, with a specific emphasis on examining the microstructural evolution during deformation and its relation to the scatter in strength at finer sizes.

2. Experimental material and procedure

Commercially pure nickel wire (~99.994%) with a diameter of ~115 μm was procured from Alfa Aesar. Smaller diameter microwires were produced by electropolishing the as-received wires in a mixture of ethanol and perchloric acid (9:1 volume ratio), at a temperature of 253 K; wires with diameter < 20 μm were prepared at 243 K. Wires of uniform diameter were obtained by electropolishing in a horizontal mounting setup with a nickel anode and a constant current pulsed-power source.

A single frame bench top universal testing machine (ADMET 560X series), mounted on an optical bench, was used in a horizontal configuration for the tensile testing of microwires. Electropolished Ni wires were sandwiched between two aluminum plates with a cyanoacrylate adhesive ("Fevi Kwick"), which acted as grips for subsequent tensile testing. For ease of handling and mounting on the machine, the whole testing assembly of wire and grips was mounted on a paper sheet. The paper sheet was cut in the gage section before starting the test. Nickel wires with a nominal gage length of 3 mm were tested at a nominal strain rate of $5.5 \times 10^{-4}\ s^{-1}$. A 20 N capacity load cell from Futek Advanced Sensor Technology was used for better resolution. The fracture strength of samples that failed in the gage section was considered for the present study to minimize issues with displacement measurements and sample misalignment. The nominal strains were calculated from the lead screw displacement, but this included machine compliance as noted from an experiment that measured strain directly on the specimen using digital image correlation (DIC) on a cylindrical wire sample that was polished to obtain a flat surface.

A scanning electron microscope (SEM), Quanta-FEI, was used to measure the diameter of microwires. The fracture surfaces of selected samples were also examined by SEM. A Sirion-FEI SEM equipped with electron back scatter imaging from TSL-OIM was used for microstructural characterization. The as-received wires were mechanically polished with diamond pastes and subsequently electropolished at 253 K with a 20 V constant DC supply for EBSD measurements. Focused ion beam milling was used to prepare longitudinal-sections near the fracture tips for EBSD. A probe current of 11.5 nano amperes (nA) was used initially, followed by ion polishing at 2.5 nA and 1 nA current to obtain a smooth surface. A step size of 40 nm was used for EBSD scans. A grain tolerance angle of 5 degrees, a confidence index of 0.1 and a minimum grain size of two pixels with multiple rows were used for identifying grains.

3. Results

3.1. Initial microstructure

Fig. 1 shows EBSD micrographs along a longitudinal section near the surface and center of the as-received microwires. The grains were elongated along the wire axis with a longitudinal grain size of ~8 to 14 μm and a transverse grain size of 100–250 nm. The inverse pole figure maps along the wire axis showed a strong < 111 > fiber texture which increases from surface (15 times random) to the center of wire (26 times random). Table 1 lists the grain boundary character distribution for the wires used in the present study and in an earlier study by Warthi et al. [18]; the fraction of coincident site lattice (CSL) boundaries is also given. Note that there is a slight variation in the grain boundary character at the surface and in the interior region. Also, although the materials used in the two studies were obtained from the same source, there are clear differences in the grain boundary character distribution, apart from the variations in grain size along and transverse to the drawing axis.

3.2. Strength

Fig. 2a shows some typical engineering stress strain curves for the microwires, with the strength ranging from a low of ~900 MPa to a high of ~1500 MPa. The variation in initial slopes with wire diameter was a consequence of machine compliance and gripping issues, as the lead screw displacement was used for calculating engineering strain. Digital image correlation (DIC) can be used for local strain measurements on the specimen, but this typically needs flat surfaces. Therefore, a 115 μm wire was slightly mechanical polished to produce a flat surface, lightly electropolished and sprayed with 10 μm alumina particles before tensile testing. Fig. 3 provides images recorded during a tensile test on such a specimen, illustrating the initiation of necking. The engineering stress–strain curves based on the overall lead-screw displacement of the tensile setup and the local strains from DIC, given in the *Supplementary* Fig. S1, showed substantial differences, indicating that the strains recorded from the lead-screw displacements were not equivalent of deformation within the specimen gauge length.

In view of the limited nominal tensile ductility, the engineering fracture strength is considered for further analysis. All of the experimental data in terms of wire diameter (Φ) and fracture strength are summarized in the Supplementary Table ST1. The effect of wire diameter on the fracture strength is shown in Fig. 2b. The deformation and fracture behavior can be classified broadly into two regimes: (a) regime 1, for $\Phi \le 50$ μm exhibiting a large scatter in strength and (b) regime 2, for $\Phi > 50$ μm, displaying limited scatter.

It is appropriate to consider a Weibull approach to strength in view of the considerable scatter and limited ductility at finer wire sizes. The cumulative probability for failure of a material (P_F) at a given stress σ can be expressed with a Weibull approach as [2]

$$P_F(\sigma, V) = 1 - \exp\left(-V\left(\frac{\sigma}{\Sigma_{V0}}\right)^{m_V}\right) \tag{1}$$

where $P_F=(i\text{-}0.5)/n$, with n being the total number of samples tested, i is the sample rank in ascending order of failure stress, V is the volume of sample, m_V is the Weibull modulus and Σ_{V0} is the scaling parameter. Fig. 4 shows the cumulative failure probability and a double logarithmic Weibull plot for both

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