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Structure-sensitive properties during room-temperature wire drawing at various speeds in nickel 200

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The effects of drawing speed, cell size and grain size on the yield strength of nickel 200 wires drawn at room temperature up to a true strain of 2.09 have been investigated. The wire drawing speeds in the range from 17 to 140 mm s^{-1} do not show any effect on the yield strength, cell size and grain size of drawn wires. However, the cell sizes as well as grain sizes decrease with increase in true wire drawing strain when their values are averaged over all the wire drawing speeds at a given strain. Even though the Hall–Petch equation is valid for all the grain diameters observed in this study, the graph suggests that two distinct linear regimes may be more appropriate to properly describe the strengthening mechanisms during wire drawing. The cell diameter has been correlated with the yield strengths of drawn wires by an inverse relationship.

1. Introduction

The control of microstructures to improve the processing of drawn wires in various metals has been a subject of study for quite some time. However, the details of microstructural parameters taken into consideration are usually limited to cell sizes, dislocation density, misorientation angles etc. during large-strain plastic deformational studies. The variation of grain sizes with yield strength is a portion of research in this field which has been quite neglected. The purpose of this paper is to report the combined effect of grain size, cell size and wire drawing speed on the strength of nickel 200 during room-temperature wire drawing. It must also be noted that large-strain plastic deformational study on nickel has so far been reported only for the case of rolling [1] up to a true thickness strain of 6.

Besides room-temperature rolling of nickel [1], the published studies involving large plastic strains in pure metals include wire drawing of Al [2], Cu [3], Nb [4] and Fe [5]. An attempt has been made in each of these cases to determine the relationship between cell size and yield strength without any regard to changes in grain sizes. However, the Hall–Petch relationship [6, 7] has been generally used to describe the grain boundary strengthening as follows:

$$\sigma = \sigma_0 + KD^{-m} \tag{1}$$

where σ is the flow stress (usually the yield strength), σ_0 and K are the materials constants and D is the grain diameter. The exponent m is generally believed to be 1/2, even though exceptions do exist [8]. This would mean that a linear relationship between yield strength and $D^{-1/2}$ is to be expected. However, it has been observed that two separate linear relationships between σ and $D^{-1/2}$ are required to cover the entire range of grain sizes in 316L stainless steel [9], nickel [10] and aluminium [11]. One of the explanations given by Young and Sherby [12] is that for sizes greater than 0.4 μm the grain-size strengthening dominates in iron while the cell-size strengthening should be dominating for sizes less than 0.4 μm. Another explanation suggested by Thompson and Saxton [13] includes the concept of reaching grain-boundary saturation with decreasing size to the extent that eventually cell-size strengthening will be the controlling mechanism. Also, the limited grain size range in which the Equation 1 is valid was shown by Thompson *et al.* [14] in aluminium, copper and brass.

The decrease in cell size during room-temperature wire drawing deformation has been reported for Fe [15], Al [2] and Cu [3]. The cell sizes have been related to yield strengths of wires with the help of the following equation:

$$\sigma = \sigma_{01} + K_1 d^{-1} \tag{2}$$

where σ is the flow stress, d is the cell size and K_1 and σ_{01} are constants. Implied in this equation is the fact that the cell- as well as the grain-size strengthening is incorporated. The details of wire drawing characteristics of nickel have not been reported in the literature and this paper examines the effect of wire drawing speed, cell size and grain size on the yield strength of drawn wires of nickel 200.

2. Experimental procedure

The spectrographic analysis of nickel 200 used in this study is shown in Table I. Rods of 9.25 mm diameter

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TABLE I Spectrographic analysis

Element	C	Mn	Si	S	Fe	Ni
Content (wt %)	0.070	0.140	0.010	0.003	0.030	Bal.

were annealed at 850 °C for 4 h prior to the wire-drawing deformation process. These rods were drawn at room temperature using four different speeds (139.7, 84.7, 42.3 and 16.9 mm s^{-1}) to various diameters with the help of dies, which would allow a 20% reduction in area per pass, to a final diameter corresponding to a true wire drawing strain of 2.09. The yield strength of the wires was determined by the 0.2% offset method using an Instron tensile testing machine at a crosshead speed of 1.27 mm min^{-1}. The grain size measurements were made by standard metallographic procedures on the short cross-sections of the wires and the samples were etched with aqueous solutions consisting of 10 wt % ammonium persulphate, $(NH_4)_2S_2O_8$, and 10 wt % sodium cyanide, NaCN, mixed in equal proportions. The grain diameters were calculated by multiplying the intercept length by 1.68.

Thin slices of 0.5 mm thickness were cut from the short cross-sections of the wires with the help of a Buehler Isomet slow-cutting machine. The wafers were hand-ground to a thickness of 0.25 mm and then polished in a Tenupol-2 electropolishing unit until perforation. The polishing conditions included a temperature of − 20 °C, a voltage of 11 to 13 V and a flow rate of 10; the solution consisted of methanol and nitric acid in the ratio of 3:1. The microstructures were observed in a Hitachi H-8000 electron microscope at 200 kV. Nearly 20 pictures were taken at 30 000 magnification for each wire, using at least five different foils, for the determination of cell sizes. The cell sizes were determined with the help of the linear intercept method and the intercept lengths were multiplied by 1.68 to obtain cell diameters. A minimum of 500 intersections of cell boundaries with the test line was used to determine the average intercept length.

3. Results and discussion

Fig. 1 shows the effect of true wire drawing strain on yield strength values of nickel 200 wires at various wire drawing speeds, up to a maximum strain value of 2.09. It can be readily seen from this figure that the effect of wire drawing speed on the yield strength of the drawn wires is almost negligible for the range of true wire drawing strains used in this study. However, there is a definite increase in yield strength as a function of true wire drawing strain. In order to understand the details of this latter observation, Fig. 2 has been drawn to show the variation of yield strength with true wire drawing strain when the yield strength values for various wire drawing speeds at a given strain are averaged. The work-hardening envelope from this figure indicates that the initial hardening takes place at a decreasing rate up to a true strain of nearly 0.9. Beyond this strain a linear hardening has been observed in this research. The linear hardening rate is 116 MN m^{-2} per unit strain, as determined by

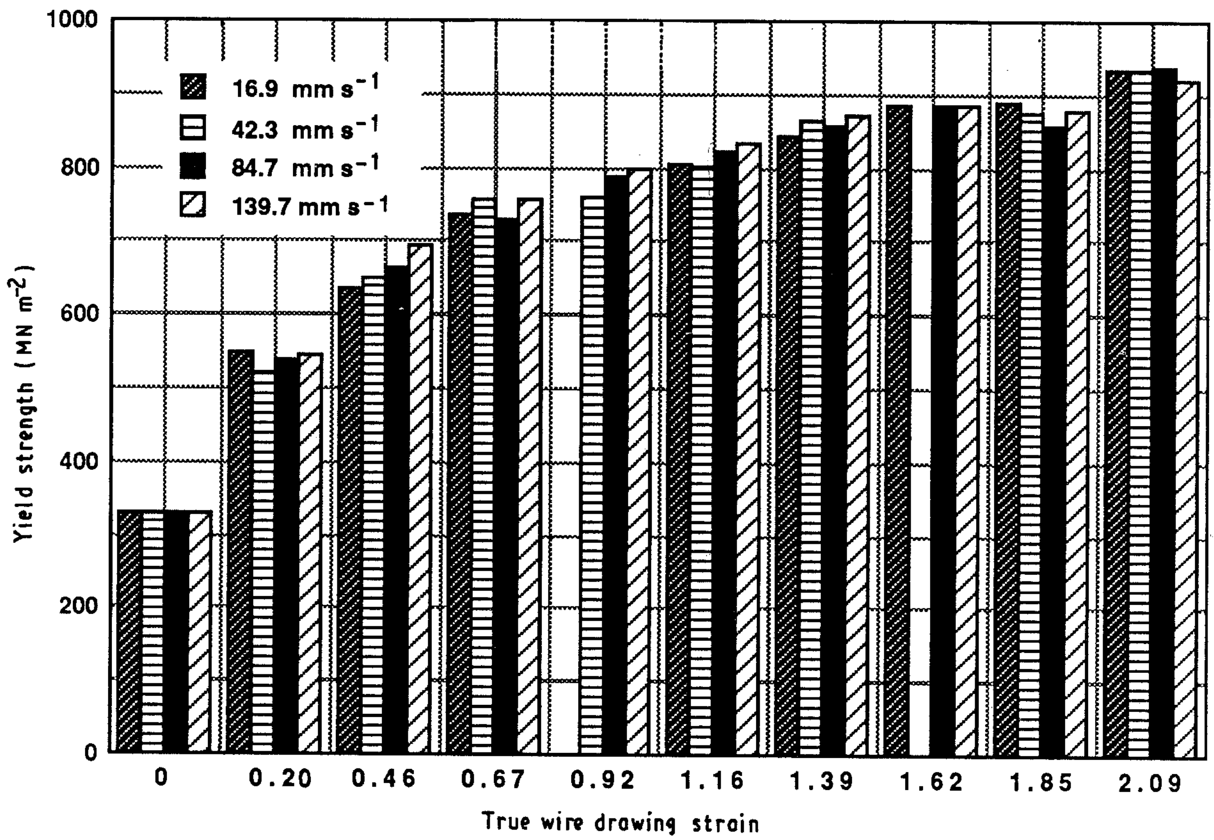

Figure 1 Histogram showing the effect of wire drawing speed on the values of yield strength of nickel 200 wires at various true wire-drawing strains.

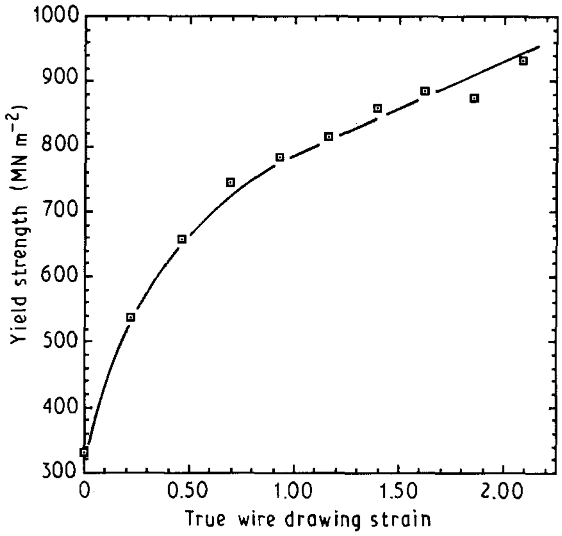

Figure 2 Yield strength versus true wire drawing strain for nickel 200.

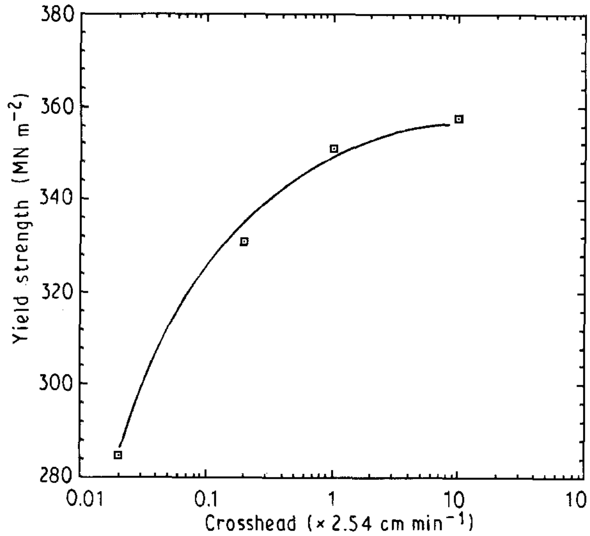

Figure 3 Variation of yield strength and ultimate tensile strength as a function of crosshead speed (it can be converted into strain rate by dividing the values by the gauge length of the specimens, which was 50.8 mm in this study). UTS = 365 MN m^{-2}.

linear regression analysis of the data points (with a correlation factor of 0.959). The value of this strain-hardening modulus lies intermediate between those of cold drawn wires of Fe [15] and Cu [16] with values of 141.3 and 51.7 MN m^{-2}, respectively.

The insensitivity of wire drawing speed towards the yield strength of drawn wires of nickel 200 in this study led us to explore the strain sensitivity during tensile testing. A 9.25 mm (annealed) rod of our nickel was tested in an Instron tensile testing machine in a range of crosshead speeds from 0.02 to 254 mm min^{-1} in order to determine the yield strength and ultimate tensile strength. Fig. 3 shows the variation of yield strength with crosshead speed (it can be converted to strain rate by dividing by the gauge length of the specimen which was 50.8 mm in this study) and the change in yield strength values from 285 to nearly 355 MN m^{-2} in the indicated strain-rate regime is clearly evident. Thus we conclude that the range of wire drawing speed was, perhaps, too narrow to show the strain-rate effect on the yield strength of drawn wires. It must also be noted that for some unknown reason the ultimate tensile strength (365 MN m^{-2}) was found to be totally insensitive to the changes in crosshead speed in this experiment.

The grain sizes were measured on the short cross-sections of the drawn wires of this study and Fig. 4 shows the variation of grain sizes with true wire drawing strain. A monotonic decrease in the grain size is clearly seen in this figure. A correlation between the grain size and yield strength of nickel 200 drawn wires was made with the help of the well established Hall–Petch equation. Fig. 5 shows a graph of the yield strength of the wire and the inverse square root of the grain diameter. No curve has been drawn (intentionally) in this graph. It is quite obvious that a single linear relation between the two parameters would not be valid here. We speculate that the data can be best fitted with the help of two straight lines, joined together with the help of a curve showing a smooth transition from region 1 (towards larger grain diameters) to region 2 (towards smaller grain diameters).

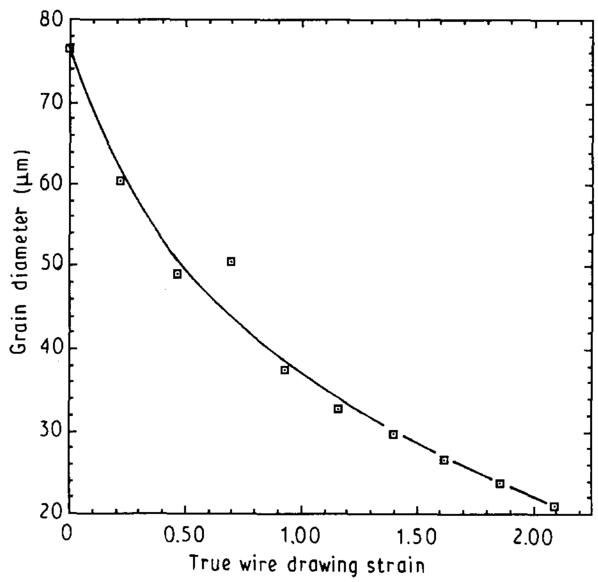

Figure 4 Variation of grain diameter with true wire-drawing strain for nickel 200.

It must be noted that a graph of this kind is not expected to show an exclusive grain-size dependence on yield strength of drawn nickel 200 wires, because grain diameters are not the only microstructural parameters that affect the yield strength values. However, it is interesting to find that the concept of dividing the graph (Fig. 5) in two separate regions in nickel has also been reported in the literature: Thompson [10] used recrystallized grains (which is the most appropriate kind for comparison with the cold-worked grains used in this study) to exclusively determine the grain-size effect on yield strength in nickel. However, the interesting comparison lies in the fact that both studies (this and that of Thompson [10]) indicate a higher slope for larger grains and lower slopes for smaller grains. It must also be noted that for a given diameter the yield strength of nickel reported by Thompson [10] is much lower than the yield strength observed in

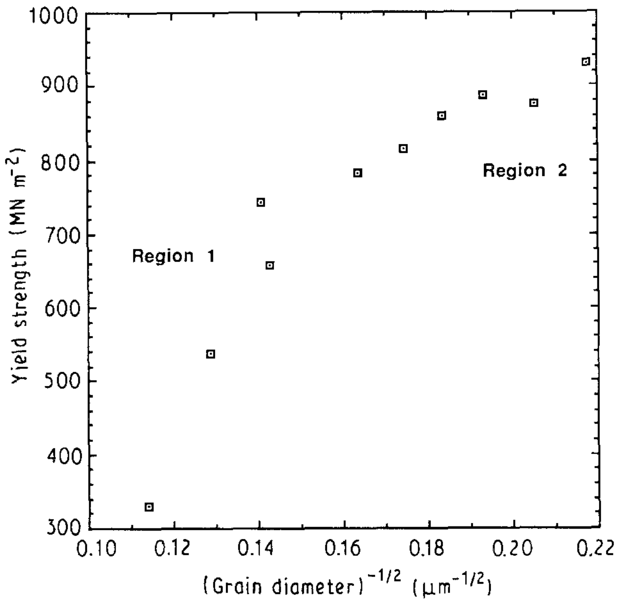

Figure 5 Variation of yield strength with the inverse square root of grain diameter (Hall–Petch plot).

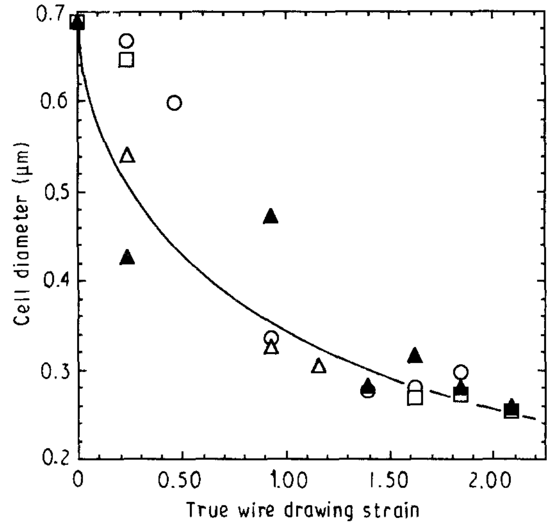

Figure 7 Variation of cell diameter with true wire- drawing strain at various wire drawing speeds: (○) 139, (□) 85, (△) 42, (▲) $17\ \mathrm{mm\,s^{-1}}$.

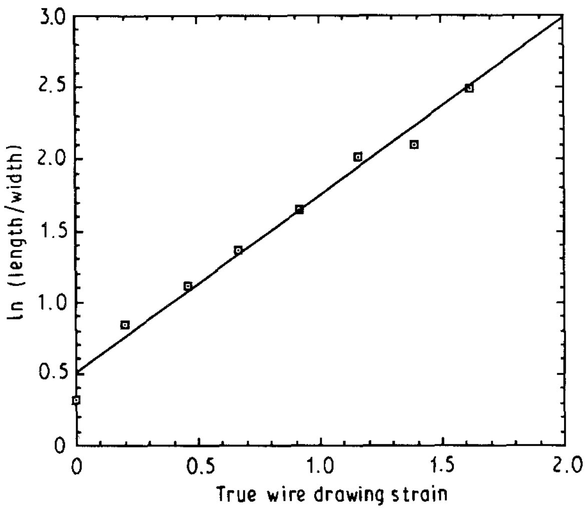

Figure 6 Variation in aspect ratio of grains with true wire drawing strain in nickel 200 wires.

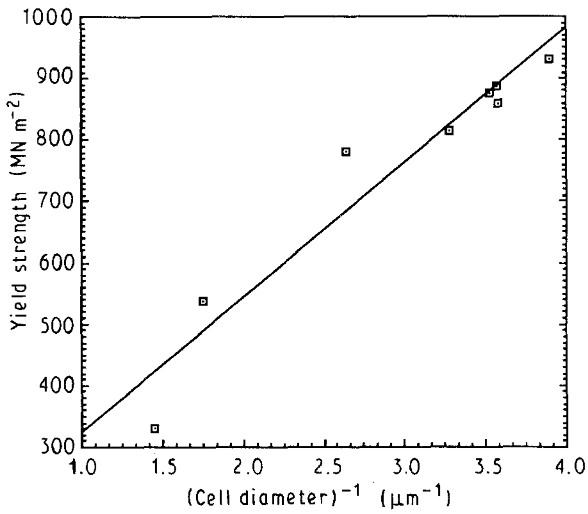

Figure 8 The correlation between the yield strength and inverse of cell diameter for nickel 200 drawn wires. Slope = $219\ \mathrm{MN\,m^{-2}\,\mu m}$.

this study. The difference can be attributed to the fact that Thompson used recrystallized grains rather than the highly deformed grains of this study. We speculate that the region 1 is dominated by grain-size strengthening while region 2 may actually involve cell-size strengthening as well (note that the yield-strength dependence on cell size would use an exponent of -1 rather than $-1/2$) as shown in Fig. 5.

One of the measures of anisotropy in metals is the variation in length to width ratio of the grains during the deformation process. Fig. 6 shows this variation as a function of true wire drawing strain. It has been found that the data can be fitted very accurately if the natural logarithm of the length to width ratio of the grains is plotted against true wire drawing strain. Thus a quantitative description indicates that the length to width ratio of grains increases exponentially with true wire drawing strain.

Fig. 7 shows the variation of cell size with true wire drawing strain at four different wire drawing speeds in the nickel 200 of this study. This figure shows that there is no systematic variation of cell diameter as a function of wire drawing speed. It must be noted that the differences in the cell diameter at various wire drawing speeds at a given true wire drawing strain decrease with increase in true wire drawing strain. This may be explained on the basis of the increase in the extent of dynamic recovery taking place in finer wires leading to a balance between the increase in total dislocation density, resulting in smaller cells, and the dynamic recovery, resulting in either increasing or stabilizing cell size. However, Fig. 7 shows that the average cell diameter (cell diameters averaged for the four different wire drawing speeds at a given true wire drawing strain) decreases consistently with true wire drawing strain as shown by the curve drawn in this figure. Thus it is possible to relate the cell diameter to yield strength with an inverse proportionality between the two as shown in Fig. 8. It is quite interesting to find that the slope of the line in Fig. 8 has been found to be 219 $MN\,m^{-2}\,\mu m$, which is higher than the values reported for both Cu [17] and Al [2] with values of 80 and 16 $MN\,m^{-2}\,\mu m$, respectively.

4. Conclusions

1. There is no effect of wire drawing speed, in the range from 16.9 to 139.7 $mm\,s^{-1}$, on the yield strength, cell size and grain size of drawn nickel 200 wires.

2. It appears that a typical Hall–Petch plot for nickel consists of two separate linear portions, irrespective of whether recrystallized or highly cold-worked grain diameters are used. The higher slopes for smaller grain diameters in this plot may be dominated by grain-boundary strengthening, while smaller slopes for smaller grain diameters may be attributed to the dominance of cell-boundary strengthening.

3. The cell-size strengthening for nickel 200 wires can be described by plotting a graph between yield strength and inverse cell diameter (averaged for the various wire drawing speeds at a given true strain).

4. The range of selected wire drawing speed was too narrow to exhibit strain-rate sensitivity of the yield strength. However, a much broader range of strain rates provided by the tensile testing experiment confirms that the yield strength is strain-rate sensitive.

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