THE EFFECT OF GRAIN SIZE ON THE DEFORMATION RESISTANCE OF NICKEL

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The effect of the grain size l of commerical nickel on the lower yield point, σ_y, and flow stress, σ_f, has been investigated. From the relationship between σ_y and $l^{-1/2}$ and between σ_f and $l^{-1/2}$, and also by extrapolation, the parameters $\sigma_i(\sigma_i^f)$ and $k_y(k_f)$, which occur in Petch's well-known expression, were determined. It was found that the values of these parameters depend on the previous history of the samples. It is suggested that the more marked dependence of the deformation resistance of nickel on grain size arising from certain thermal treatments is due to the segregation of impurities to the grain boundaries. It is shown that this is in accord with the presence of grain-boundary hardening and with its dependence on quenching temperature.

Hall [1] first showed that the relationship between the lower yield point, σ_y, of low-carbon steel and the grain size, l, is expressed by the equation

$$\sigma_y = \sigma_i + k_y l^{-1/2}. \qquad (1)$$

Many authors have since confirmed this relationship [2–11 and others].

The interpretation of Eq. (1) suggested by Hall [1] and Petch [2] is based on the following two assumptions: (1) that planar pile-ups of dislocations occur which are blocked by grain boundaries; (2) transmission of deformation at the lower yield point from grain to grain takes place as a result of rupture of intergrain boundaries by the dislocation pile-ups. From this point of view, the dependence of the lower yield point on the grain dimensions is completely determined by grain-boundary strength, a measure of which is given by the parameter k_y.

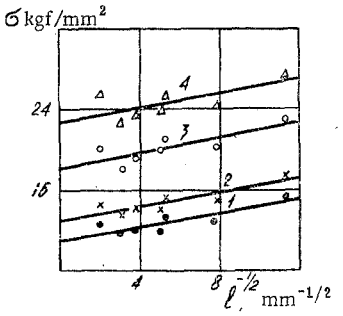

Fig. 1. The relationship between deformation resistance of nickel quenched from 700° in water and grain size. 1) $\varepsilon = 0.5\%$; 2) $\varepsilon = 1.0\%$; 3) $\varepsilon = 2.5\%$; 4) $\varepsilon = 4.0\%$.

Later, Cottrell [12] gave a different interpretation of Eq. (1). He suggested that deformation is transmitted from grain to grain by the excitation of Frank-Read sources in neighboring grains under the action of a stress which is the sum of the applied stress and an additional stress due to the pile-up of dislocations at the grain boundary. In this case the parameter k_y characterizes the degree of blocking of Frank-Read sources by carbon-nitrogen interstitials.

Subsequently Petch also used the pile-up model [5]. However, he now [13] takes a somewhat different view, assuming that the excess stress in adjacent grains is created by a slip band, not necessarily consisting of a planar dislocation pile-up that has approached.

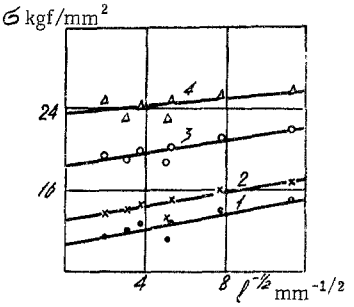

Fig. 2. The relationship between the deformation resistance of nickel water-quenched from 700° and then heated for 10 min at 180°, and the grain size: 1) $\varepsilon - \varepsilon_L = 0.5\%$; 2) $\varepsilon - \varepsilon_L = 1.0\%$; 3) $\varepsilon - \varepsilon_L = 2.5\%$; 4) $\varepsilon - \varepsilon_L = 4.0\%$.

Recently, many experimental results have been obtained [14–21] that do not fit the framework of the above theories. Theories based on the pile-up mechanism may now be considered to have been refuted, since neither in α-iron and low-carbon [17,18,21] nor in other bcc metals [19,20], which also have yield points due to interstitials, have planar dislocation pile-ups been detected. The view, contrary to Cottell's assumption, that in the process of plastic deformation dislocations pinned by interstitials do not break away but remain stationary is becoming more and more widely accepted [21–23], the presence of a yield point and a yield plateau being linked either with an initial by small number of free dislocations [22,23] or with the generation of dislocations at interfaces such as grain boundaries or interfaces between inclusions and matrix [21]. The latter assumption is supported by the results of [20,24,25].

Our limited understanding of the yield mechanism naturally results in a lack of agreement as to the physical processes which determine the parameter k_y. In this respect the phenomenon of forced slip at the grain boundaries discovered by Johnson and Shaw [26] is of importance. Since a Lüders-Chernov

Table 1

T_0, °C	700	750	800	850	900	1000	1150
l, mm	0.008	0.015	0.030	0.038	0.070	0.104	0.200
$l^{-1/2}$, mm$^{-1/2}$	11.3	7.8	5.3	5.1	3.8	3.1	2.0

line, propagated at the lower yield point, must overcome these hardened areas around the grain boundaries, according to Johnson [27] the effect of grain size on the parameter σ_y will be the greater, the higher the rate of hardening and the stronger the blocking of the dislocations.

Recently, Li [28] suggested a new theory, on the basis of which he derived a quantitative expression for k_y. Li introduced the concept of new dislocation sources formed by jogs at the grain boundaries. He assumed that yielding begins when the external stress causes production of dislocations at these sources. On the basis of this model the following expression for the parameter k_y is obtained:

$$k_y = \alpha\mu b\,(8\,m/\pi)^{1/2}, \qquad (2)$$

where α is of the order of 0.4, **b** is the Burgers vector, and m is the density of grain boundary sources (jogs at the grain boundaries), which according to Li's theory depends on the temperature and the concentration of impurities.

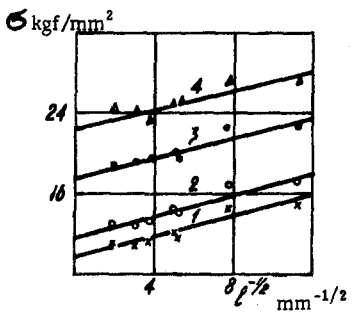

Fig. 3. The relationship between deformation resistance of nickel water-quenched from 600° after slow cooling (50°/hr) from T_0 to T_k and grain size: 1) $\varepsilon = 0.5\%$; 2) $\varepsilon = 1.0\%$; 3) $\varepsilon = 2.5\%$; 4) $\varepsilon = 4.0\%$.

Many investigations [13,14, and others] have shown that the relationship between the flow stress and the grain size is similar to (1):

$$\sigma_f = \sigma_i^f + k_f l^{-1/2}. \qquad (3)$$

Usually for α-iron and low-carbon steel k_f is less than k_y [13], although according to the results of Conrad and Schoek [14] $k_f = k_y$.

In the paper mentioned above [28], Li also considered the effect of plastic deformation on the parameter k_f, and showed that if, in the deformation of any type of material, recovery occurs, k_f must decrease with increasing deformation. In the absence of recovery k_f must increase with increasing deformation. It should be noted that (1) and (3) are valid not only for bcc metals and alloys, but also for those with fcc and hexagonal ones [13].

The stresses σ_i and σ_i^f in Eqs. (1) and (3) respectively represent the resistance to movement of the free dislocations inside the grains due to the Peierls-Nabarro force, inclusions, dissolved impurity atoms, and dislocations.

In summary it may be said that the problem of the effect of grain size on the mechanical properties of materials is still far from solved, and therefore any new data will undoubtedly be of value. The present paper is devoted to an investigation of the effect of grain size on the deformation resistance of commercial nickel (type N1). Investigation of nickel is of particular interest since, like α-iron, it is subject to rapid deformation aging, caused by the presence of carbon [29–34].

MATERIAL AND EXPERIMENTAL PROCEDURE

Samples of grade N1 nickel were prepared from wire 1 mm in diameter. The nominal length of the samples was 50 mm. The grain size was varied by vacuum annealing for 1 hr at various temperatures. To produce the largest grain size the samples were annealed in a sealed tube. The annealing conditions and the grain sizes obtained are listed in Table 1.

After annealing the samples were submitted to the heat treatments detailed in Table 2. The samples were tested at room temperature on tensile testing machine [35]. The extension diagrams were recorded photographically. The rate of deformation was 60%/hr. Each experimental point is the mean of values obtained for 5–10 samples. On all the graphs the straight lines relating σ_y or σ_f and $l^{-1/2}$ were plotted by the method of least squares.

RESULTS

The experimental results obtained are given in Table 2 and in Figs. 1–6. From Fig. 1 it will be seen that in the case of samples quenched in water from 700° C after rapid cooling from the annealing temperature T_0, for the deformations $\varepsilon = 0.5\%$, 1.0%, 2.5%, and 4.0% the relationship between $l^{-1/2}$ and the resistance to deformation of is more or less linear. For higher deformations in this and in the remaining series of experiments it was observed that on the one hand the scatter of the

Heat treatment after annealing	ε, %	σ_i, kg/mm^2	k, kg/mm$^{3/2}$
Rapid cooling from T_0 to 700° C, quenching from 700° C in water.	0.5	11.0	0.34
	1.0	13.0	0.35
	2.5	18.2	0.38
	4.0	22.7	0.35
Rapid cooling from T_0 to 700° C, quenching from 700° C in water, aging at 180° for 10 min	$\varepsilon - \varepsilon_L = 0.5$	10.5	0.37
	$\varepsilon - \varepsilon_L = 1.0$	13.0	0.33
	$\varepsilon - \varepsilon_L = 2.5$	18.4	0.30
	$\varepsilon - \varepsilon_L = 4.0$	23.6	0.16
Slow cooling at 50°/hr from T_0 to T_k	lower yield point	4.4	0.97
	$\varepsilon - \varepsilon_L = 0.5$	9.2	0.76
	$\varepsilon - \varepsilon_L = 2.0$	15.6	0.73
Slow cooling at 50°/hr from T_0 to T_k, soaking at 600° for 1 hr, followed by quenching in water.	0.5	9.6	0.52
	1.0	11.7	0.51
	2.5	17.4	0.50
	4.0	22.2	0.49
Slow cooling at 50°/hr from T_0 to T_k, soaking at 500° for 1 hr followed by quenching in water.	$\varepsilon - \varepsilon_L = 0.5$	9.4	0.49
	$\varepsilon - \varepsilon_L = 1.0$	12.0	0.43
	$\varepsilon - \varepsilon_L = 2.5$	17.7	0.39
	$\varepsilon - \varepsilon_L = 4.0$	22.7	0.32

experimental points was very high, and on the other hand the deformation resistance of the samples with the largest grains was anomalously high. The latter effect is apparently due to the special conditions used to produce the large grains. Consequently no conclusions can be drawn about the nature of the relationship between the deformation resistance of nickel and the grain size at large deformations.

From Table 2 it will be seen that k_f is practically independent of ε and has a mean value of 0.35 kg/mm$^{3/2}$. Of course, σ_i^f increases with increasing deformation as a result of work hardening of the material. Since the samples were quenched from the relatively high temperature of 700° C, it was to be expected that the yield phenomenon would not be observed. This was in fact true, with the exception of the samples with the smallest grain size, which exhibited a very indistinct yield plateau.

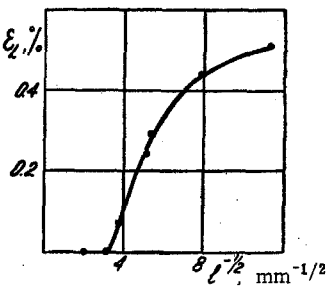

Fig. 4. The relationship between the Lüders-Chernov deformation and the grain size of samples slowly cooled from T_0 to T_k (50°/hr).

The second batch of samples water quenched from 700° C immediately after grain growth and rapid cooling to this temperature was submitted to an additional thermal treatment—aging at 180° C for 10 min. If the quantity of carbon which can segregate at the dislocations under the above conditions is calculated by the Cottrell-Bilby formula [36] from the data of [34,37], it is found that the saturation of the atmosphere may be up to two-thirds of the possible maximum. Thus in this case blocking of the dislocations should have a considerable effect. The experiments showed that the additional aging treatment did in fact somewhat change the behavior of the material, but only in the case of the samples with the two smallest grain sizes. While a slight yield plateau was observed in the unaged material only in the case of samples with the smallest grain size, aging not only increased the yield effect in these samples but also produced a yield plateau in the samples with grain size l = = 0.015 mm. In both batches the plateaus were sufficiently well defined for the parameters k_y and σ_i to be determined extrapolation (see below). It was found that k_y = 0.30 kg/mm$^{3/2}$ and σ_i = 6.8 kg/mm^2.

The values of k_f and σ_i^f obtained for various deformations (Fig. 2) [for l = 0.008 and 0.015 mm the values were determined from the end of the yield plateaus (see below)] are listed in Table 2. It will be seen that, for small values of ε, k_f is very close to k_y, determined by extrapolation, and also to the values of k_f obtained in the first set of experiments. It is of particular interest that k_f decreases by a factor of more than two as ε is increased from 0.5% to 4.0%.

The samples which had been cooled slowly (50°/hr) and then quenched in water from 600° C behaved quite differently (Fig. 3). This thermal treatment resulted in an increase in the parameter k_f and in a decrease in σ_i^f. The samples quenched from 500° (Table 2) behaved similarly, although in this case the parameter k_f decreased significantly as ε increased from 0.5 to 4.0%.

In the slowly cooled samples the dislocations were, of course, strongly blocked by the carbon atmospheres (or even superatmospheres [38]), which resulted in the appearance of a yield plateau on the extension of all the samples except those with the two largest grain sizes. The relationship between the size of the yield plateau (Lüders-Chernov deformation) and $l^{-1/2}$ is shown in Fig. 4, from which it will be seen that in this case ε_L decreases rapidly with increasing grain size.

It has been shown [19,39,40] that the extrapolation method [12] of determining the parameters k_y and σ_i gives excellent results which agree with those obtained from the relationship between k_y and $l^{-1/2}$.

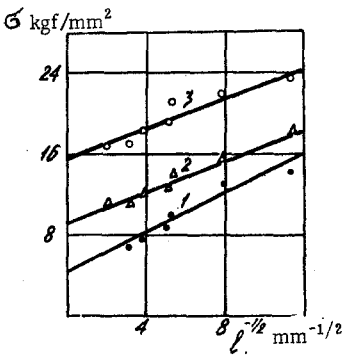

Fig. 5. The relationship between the lower yield point and flow stress of nickel cooled at the rate of 50°/hr and the grain size: 1) lower yield point; 2) $\varepsilon - \varepsilon_L$ = 0.5%; 3) $\varepsilon - \varepsilon_L$ = 2.0%.

The importance of the extrapolation method lies in the fact that it indicates whether the parameters k_y and σ_i vary with the grain size, and thus tests the validity of using samples with various grain sizes to determine k_y and σ_i. Therefore the extrapolation method was used in addition to the grain-size technique (Fig. 5). The values of $k_y l^{-1/2}$ determined by the extrapolation method are represented by the points on Fig. 6. It will be seen that the points lie satisfactorily close to the straight line 1, which was plotted from the values of k_y determined from Fig. 5. The relationship between σ_i and $l^{-1/2}$, determined by extrapolation, is represented by curve 2 of Fig. 6; the value of σ_i determined by

the grain-size technique is given by the straight line 3. It will be seen that σ_i increases as the grain size decreases. However, since this increase is only slight, and since the value of σ_i obtained by extrapolation is close to the value given by the grain-size technique, it is probably safe to conclude that the results of the extrapolation method confirm the validity of the grain-size technique.

In plotting σ_f as a function of $l^{-1/2}$ for the slowly cooled samples it is essential to remember that σ_i is, to a first approximation, independent of the grain size, whereas the extent of the yield plateau depends closely on the grain size. Thus it can be assumed that the parameter σ_i^f will be independent of the grain size only when the deformation for which the relationship σ_f vs. $l^{-1/2}$ is plotted from the end of the yield plateau [28]. Therefore σ_f was plotted as a function of $l^{-1/2}$ for $\varepsilon - \varepsilon_L = 0.5\%$ and 2.0% (Fig. 5, Table 2). It will be seen that k_f is less than k_y, and decreases slightly as the deformation is increased.

DISCUSSION

On the basis of the above results little can be added to our understanding of the mechanism of yield phenomena. However, the results are of value in elucidating the effect of grain boundaries on deformation resistance. It appears that the results obtained can be explained on the assumption that the intensification of the grain-size effect in metals containing impurities is due to the segregation of these impurities at the grain boundaries. Such grain-boundary segregation is a well-established phenomenon [41,42].

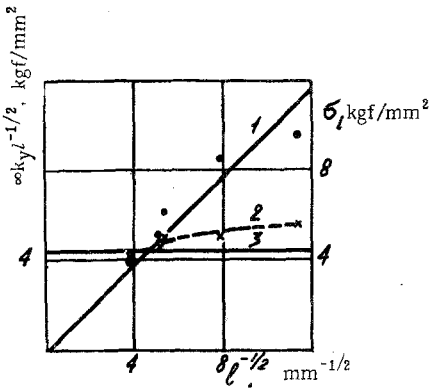

Fig. 6. The relationship between $k_y l^{-1/2}$ and σ_i determined by the two different methods, and the grain size: $k_y l^{-1/2}$: • = exptl. point—extrapolation method; 1) grain-size technique: σ_i; 2) extrapolation method; 3) grain-size technique.

It can be assumed that in the samples quenched from 700° immediately after grain growth the impurity atoms (apparently mainly carbon) will be more or less uniformly distributed throughout the samples. An additional short anneal at 180°, which results in the formation of a Cottrell atmosphere, as indicated by the appearance of a clear yield plateau during the testing of samples having grain sizes $l = 0.008$ and 0.015 mm, cannot, of course, have any significant effect on the concentration of impurity atoms at the grain boundaries. At this temperature the time necessary for noticeable segregation, evaluated by formula (5.9) of [41], is much longer than 10 min. Thus the values of the parameters k_f and σ_i^f are practically unaffected by this additional treatment, except at large deformations. However, little attention should be paid to the results of experiments in which the deformations were large, since the scatter of the experimental points was excessive.

From Table 2 it will be seen that in the case of the samples cooled slowly (50°/hr) the values of k_y and k_f are much higher, and the values of σ_i and σ_i^f much lower, than in the first two sets of experiments. We consider that the sharp increase in the parameter k is due mainly to grain-boundary hardening by intensive segregation of impurity atoms which occurs readily during slow cooling.

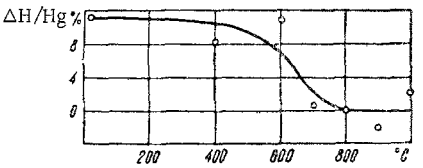

Fig. 7. The relationship between grain-boundary hardening in nickel (ΔH denotes the difference between the microhardness of the boundaries and that of the interior of the grains; h_g denotes the microhardness of the grains) and the quenching temperature.

This segregation also affects the relationship between the flow stress and the grain size and thus results in high values for k_f. The decrease in the values of σ_i produced by slow cooling is apparently due to a reduction in the concentration of impurity atoms within the grains, where they hinder the movement of dislocations.

From Table 2 it will be seen that when slowly-cooled samples were annealed at 500° and 600° for 1 hr the parameter k_f decreased and σ_i increased. However, in these cases the values of k_f were significantly higher, and the values of σ_i^f significantly lower, than the corresponding values for samples quenched from 700°. This can readily be explained by the grain-boundary segregation of impurities, which will be more marked in the former two cases than in the latter.

An additional series of experiments was carried out to confirm the grain-boundary hardening and to determine the effect on it of the last treatment conditions. In these experiments the microhardness at the grain boundaries and within the grains was measured with a PMT-3 microhardness tester, the load on the indenter being 20 g. The nickel samples used were in the form of sheets 0.5 mm thick.

To determine their initial condition they were annealed for 1 hr at 1000° and then cooled to room temperature at the rate of 50°/hr. The samples were electropolished in an electrolyte consisting of 59% H_2SO_4 and 41% H_20 (by volume) and etched in concentrated nitric acid. The microhardness was determined on samples that had been quenched from various temperatures after being held there for 1 hr. In each case not less than 40 measurements were made. The grain-boundary hardening was characterized by the ratio of the difference between the microhardness at the grain boundary and that within the grains, ΔH, to the microhardness inside the grains, H_g (which is practically independent of the quenching temperature). Figure 7 shows the variation of $\Delta H/H_g$ with quenching temperature. It will be seen that the samples in their initial condition and those quenched from 400 and 600° exhibit considerable grain-boundary hardening, but this was not observed in the samples held at higher temperatures before quenching. From a comparison of Fig. 7 with the data of Table 2 it will be seen that there is a correlation between the variation in the parameters k_f and σ_i^f and the variation in grain boundary hardening with quenching temperature: the sharp decrease in k_f and the increase in σ_i^f correspond to the rapid decrease in grain-boundary hardening between 600 and 700°. These results thus confirm the original assumption.

In conclusion the following point should be noted. In plotting the relationship between the flow stress and $l^{-1/2}$ in the present investigation the flow stresses were determined for a series of deformations measured from the end of the yield plateau. This method assumes that the "frictional" stress, σ_i, changes continuously during the transition from yield to flow. However, it is possible that in this transition σ_i increases discontinuously to the value of σ_i^f corresponding to the deformation at the end of the yield plateau. Such a discontinuity might be the result of the gradual propagation through the sample during yielding of a fixed deformation, the magnitude of which depends, in particular, on the grain size, so that underformed regions are present in the sample until the end of the yield plateau is reached. Under these conditions σ_i will express the "frictional" stress at the front of the Lüders-Chernov region. Since uniform deformation begins only after the yield deformation has been propagated through the whole sample, the latter deformation must contribute to an increase in σ_i^f. Thus in the transition from yielding to uniform flow σ_i will increase discontinuously to σ_i^f and there will be a corresponding discontinuous decrease in the parameter k. From this point of view, in determining the Hall-Petch relationship for the flow stresses, the selected constant value of the deformation must include the yield deformation.

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REFERENCES

1. E. O. Hall, Proc. Phys. Soc., **B64** 747, 1951.
2. N. J. Petch, J. Iron St. Inst., **174**, 25, 1953.
3. J. R. Low, The Structure and Properties of Metals [Russian translation], Metallurgizdat, Moscow, 1957.
4. A. Cracknell and N. J. Petch, Acta Met., **3**, 186, 1955.
5. N. J. Petch, Uspekhi fiziki metallov, 2, Metallurgizdat, Moscow, 1958.
6. N. J. Petch, Phil. Mag., **1**, 186, 1956.
7. J. Heslop and N. J. Petch, Phil. Mag., **1**, 816, 1956.
8. J. Hieslop and N. J. Petch, Phil. Mag., **2**, 649, 1957.
9. N. J. Petch, Phil. Mag., **3**, 1089, 1958.
10. J. Heslop and N. J. Petch, Phil. Mag., **3**, 1128, 1958.
11. I. Codd and N. J. Petch, Phil. Mag., **5**, 30, 1960.
12. A. H. Cottrell, Trans. Met. Soc. AIME, **212**, 192, 1958.
13. R. Armstrong, I. Codd, R. M. Douthwaite and N. J. Petch, Phil. Mag., **7**, 45, 1962.
14. H. Conrad and G. Schoeck, Acta Met., **8**, 791, 1960.
15. N. N. Davidenkov, B. I. Smirnov and V. D. Yaroshevich, FTT, **3**, 1731, 1961.
16. V. D. Yaroshevich, FTT, **3**, 3207, 1961.
17. D. G. Brandon and J. Nutting, J. Iron St. Inst., **196**, 160, 1960.
18. W. Carrington, K. F. Hall and D. McLean, Proc. Roy. Soc., **259**, 203, 1960.
19. T. C. Lindley and R. E. Smallman, Acta Met., **11**, 361, 1963.
20. L. I. Van Torne and G. Thomas, Acta Met., **11**, 881, 1963.
21. W. C. Leslie and A. S. Keh, J. Iron St. Inst., **200**, 722, 1962.
22. G. T. Hahn, Acta Met., **10**, 727, 1962.
23. W. G. Johnston, J. Appl. Phys, **33**, 2716, 1962.
24. E. Votava, Acta Met., **11**, 1105, 1963.
25. R. J. Arsenault, Acta Met., **11**, 1111, 1963.
26. A. A. Johnson and B. J. Shaw, Nature, **183**, 1542, 1959.
27. A. A. Johnson, Phil. Mag., **7**, 177, 1962.
28. J. C. M. Li, Trans. Met. Sos. AIME, **227**, 239, 1963.
29. V. F. Sukhovarov and R. P. Kharlova, FMM, **10**, 938, 1960.
30. V. F. Sukhovarov, FMM, **13**, 275, 1962.
31. V. F. Sukhovarov, N. A. Alesksandrov, and L. A. Kudryavtseva, FMM, **14**, 896, 1962.
32. V. S. Sukhovarov and L. E. Popov, FMM, **17**, no. 1, 1964.
33. L. E. Popov and V. F. Sukhovarov, FMM, **17**, no. 3, 1964.
34. V. S. Sukhovarov, FMM, **17**, no. 2, 1964.
35. L. I. Vasil'ev, FMM, **7**, 314, 1959.
36. A. H. Cottrell and B. A. Bilby, Proc. Phys. Soc., **A62**, 49, 1949.

37. P. L. Gruzin, Yu. A. Polikarpov, and G. B. Fedorov, FMM, **4**, 94, 1951.

38. R. Bullough and R. C. Newman, Proc. Roy. Soc., **A266** 109, 1962.

39. A. R. Rosenfield, J. Inst. Metals, **61**, 104, 1962–63.

40. A. R. Rosenfield and W. S. Owen, Trans. Met. Soc. AIME, **227**, 603, 1963.

41. D. McLean, Grain Boundaries in Metals [Russian translation], Metallurgizdat, Moscow, 1960.

42. V. A. Phillips, Acta Met., **11**, 1139, 1963.

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