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THE EFFECTS OF HELIUM ON HIGH-TEMPERATURE DUCTILITY OF SANDVIK 12R72HV AND INCO IN-744X

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AND MATERIALS

THE EFFECTS OF HELIUM ON HIGH-TEMPERATURE DUCTILITY OF SANDVIK 12R72HV AND INCO IN-744X

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DISTRIBUTION

This report has been distributed according to the category "Metals, Ceramics, and Materials," as given in the Standard Distribution for Unclassified Scientific and Technical Reports, TID-4500.

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ABSTRACT

Small tensile samples of Sandvik 12R72HV and Inco IN-744X were injected with helium by means of α-particle irradiation from a cyclotron to produce approximately uniform concentrations of from 1 x 10^{-6} to 2.5 x 10^{-5} atom fraction. The samples were subsequently tensile tested in vacuum between 500 and 800°C. Sandvik 12R72HV is significantly more resistant to helium embrittlement than Types 304 or 316 stainless steel. Helium in Inco IN-744X has little effect on the superplastic behavior of this alloy.

I. INTRODUCTION

Helium causes a loss of ductility in many alloys during high-temperature tensile or creep testing because it accumulates at grain boundaries where it has a strong positive influence on intergranular cracking. This has been observed in reactor irradiated samples(1, 2) and in samples that have been injected with helium by cyclotron irradiation.(3-5)

Premature intergranular failure may be due either to the joining of grain-boundary helium bubbles that have grown with the assistance of stress(6) or to the formation of grain-boundary cracks by grain-boundary sliding as promoted by helium bubbles in the grain boundary.(7) Test temperature, strain rate, and the degree of grain-boundary sliding will determine which mechanism is favored during testing. The alloys that are the subject of this paper, Sandvik 12R72HV and Inco IN-744X, are of interest because of the nature of their microstructures. Finely dispersed precipitates in the grain boundaries may be developed in Sandvik 12R72HV, whereas the super-plastic Inco IN-744X has an extraordinarily high amount of grain-boundary area per unit volume — the alloy has a grain size of only a few microns. Both of these features may be expected to mitigate the effects of helium.

II. EXPERIMENTAL PROCEDURE

The compositions of Sandvik 12R72HV and Inco IN-744X are given in Table 1. Small sheet tensile samples with a cross-section of 0.20 mm x 1.02 mm and a gage length of 12.7 mm were punched from strip and annealed as shown in Table 2. The samples were then injected with helium by means of alpha-particle irradiation from a cyclotron(7) and the resulting helium contents were determined analytically.(3)

TABLE 1

COMPOSITIONS OF ALLOYS (wt %)

Alloy	C	N	Ni	Cr	Mo	Ti	Mn	Si	Fe
Sandvik 12R72HV	0.1	-	15	15	1.2	0.5	1.8	0.5	balance
Inco IN-744X	0.06	0.03	6.6	27	-	0.2	0.4	0.5	balance

TABLE 2

ALLOY ANNEALS BEFORE TESTING

Alloy	Anneal
Sandvik 12R72HV	1 hour at 1100°C + 8 hours at 825°C
Inco IN-744X	10 minutes at 870°C

Tensile tests were performed in vacuum at a strain rate of 0.02/min. Yield strengths were measured at 0.2% elongation. Samples were held at the test temperature for one hour before the load was applied.

III. RESULTS

A. SANDVIK 12R72HV

Tensile test results are presented in Table 3 where the values shown are the average of two samples. The yield strengths of the samples that have received the higher helium dose showed an increase at 500 and 600°C which is probably due to the displacement damage caused by the irradiation. The principal effect of helium was to reduce the total elongation at 700 and 800°C; the uniform elongation remained essentially unchanged at all test temperatures.

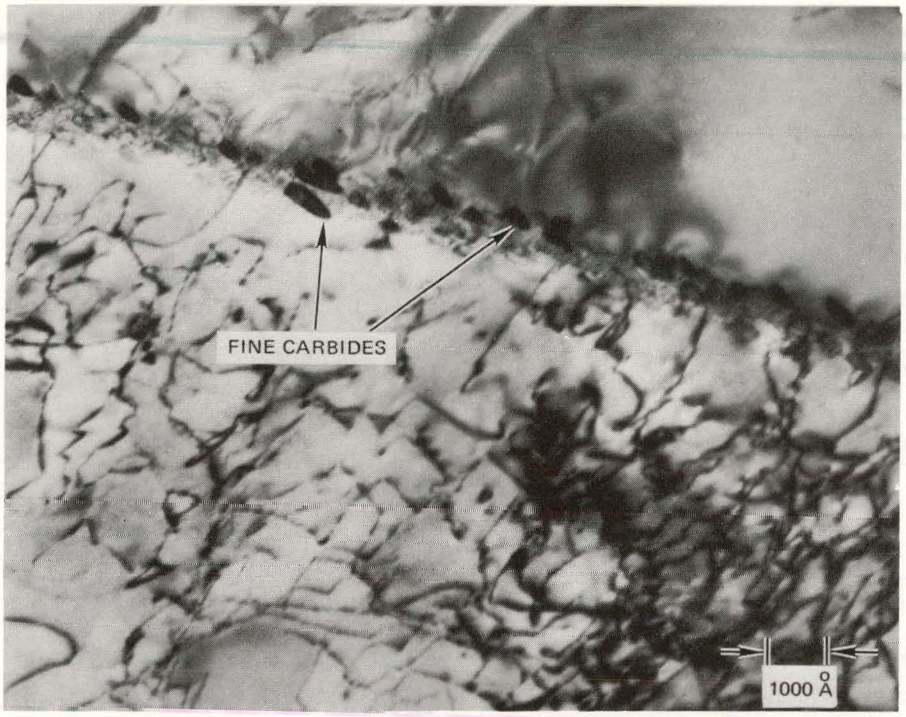
The microstructure of this alloy prior to testing consisted of austenite grains, with an average size of 35μm, with both fine and coarse $M_{23}C_6$ precipitates (as determined by electron diffraction) in the grain boundaries. The fine precipitates ranged between 100 and 200 Å in size, whereas the coarser ones were about 1μm.

Figure 1 shows these fine precipitates in a sample tested at 700°C. At this temperature, samples without helium failed transgranularly, those with the lower helium content failed in a predominantly transgranular mode, and those with the higher helium content experienced a mixed transgranular-intergranular failure. A few small helium bubbles, 20 Å in a diameter, were found attached to some of the coarse grain-boundary carbides.

At 800°C, the presence of 5×10^{-5} atom fraction helium caused the failure mode to change from transgranular to intergranular. Voids were observed in grain boundaries in the high-helium containing samples but they were not adjacent to the coarse carbide particles as shown in Figure 2. Very large helium bubbles, up to 1500 Å in size, were observed in grain boundaries (Figure 3a) and occasionally in the matrix. These large bubbles were not generally attached to carbide particles. Smaller bubbles were found throughout the matrix averaging about 50 Å in size (Figure 3b).

B. INCO IN-744X

The total elongation of this super-plastic alloy rises very rapidly with increasing temperature with a concomitant fall in strength (Table 4). The presence of helium causes the rise in total elongation to be less steep but has no effect on strength. The small increases in yield and tensile strength at 500°C are



5-12-70 UNCL 6506-4701

Figure 1. Transmission-Electron Micrograph of Sandvik 12R72HV, with 2.5×10^{-5} Atom Fraction Helium, Tested at 700°C. Fine Precipitates of $M_{23}C_6$ are Present in the Grain Boundary.

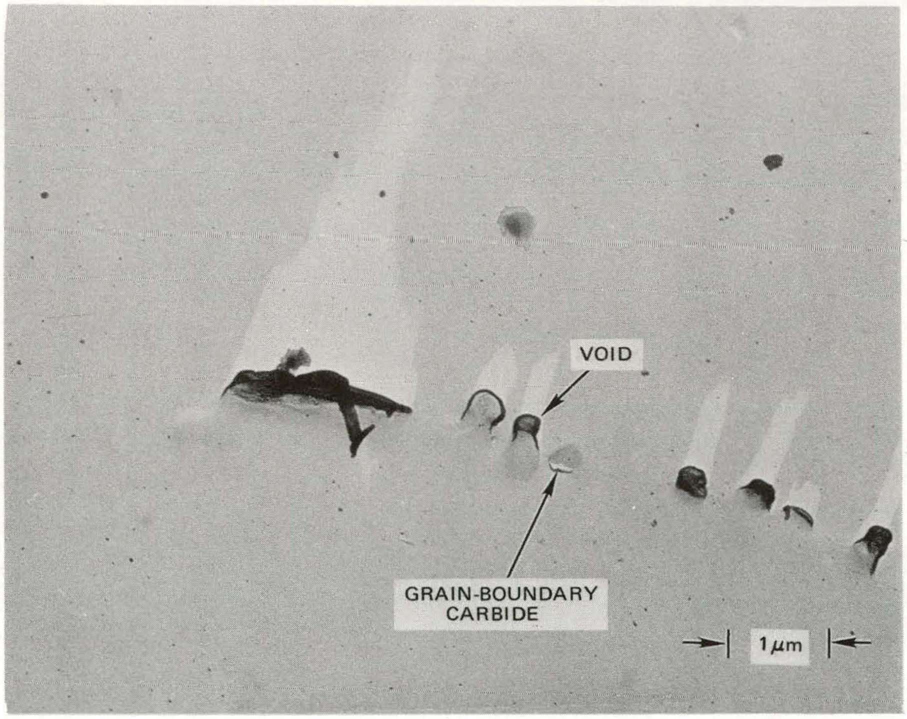


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Figure 2. Replica-Electron Micrograph of Sandvik 12R72HV, with 5×10^{-5} Atom Fraction Helium, Tested at 800°C. Voids in the Grain Boundary are not Adjacent to the Coarse Carbide Particles.

5-12-70 UNCL 6506-4703

a. Note Helium Bubbles in Grain Boundary

5-12-70 UNCL 6506-4704

b. Note Helium Bubbles in Both Matrix and Grain Boundary

Figure 3. Transmission-Electron Micrographs of Sandvik 12R72HV, with 5 x 10^{-5} Atom Fraction Helium, Tested at 800°C

TABLE 3

TENSILE TEST RESULTS FOR SANDVIK 12R72HV

Test Temperature (°C)	Atom Fraction Helium	Yield Strength (kg/mm^2)	Tensile Strength (kg/mm^2)	Elongation (%)	
				Uniform	Total
500	0	13.1	43.1	26	27
500	1×10^{-6}	13.1	41.8	25	26
500	5×10^{-5}	15.0	43.1	25	27
600	0	12.0	37.7	28	29
600	1×10^{-6}	11.7	38.5	28	30
600	5×10^{-5}	14.1	38.4	27	29
700	0	11.8	25.7	22	41
700	1×10^{-6}	11.2	25.0	21	37
700	2.5×10^{-5}	11.7	24.9	20	34
800	0	9.9	15.2	13	57
800	1×10^{-6}	10.1	15.4	14	46
800	5×10^{-5}	10.0	15.5	13	27

TABLE 4

TENSILE TEST RESULTS FOR INCO IN-744X

Test Temperature (°C)	Atom Fraction Helium	Yield Strength (kg/mm^2)	Tensile Strength (kg/mm^2)	Elongation (%)	
				Uniform	Total
500	0	52.8	56.0	2.1	18
500	1×10^{-6}	57.7	57.7	2.0	15
500	2×10^{-5}	55.1	57.9	2.0	16
600	0	26.9	28.3	2.0	53
600	1×10^{-6}	26.3	28.3	1.8	44
600	2×10^{-5}	27.1	28.4	2.0	32
700	0	11.5	14.5	3.4	205
700	1×10^{-6}	10.6	13.1	3.5	114
700	2×10^{-5}	11.5	13.8	3.2	94
800	0	3.8	4.4	3.9	>300
800	2×10^{-5}	3.5	4.2	3.5	>300

probably the residual effect of displacement damage. The uniform elongations appear merely as a formality in Table 4, since necking, in the usual sense, was absent. Rather, the entire gage length may be considered as the neck.

The microstructure of this alloy prior to testing was a mixture of austenite and ferrite grains whose size average between 1 and 2μm (Figure 4). Testing at 700°C resulted in intergranular failures for all samples without an increase in grain size. Voids and cracks were observed in grain-boundaries and they were more numerous in samples with helium. Figure 5a shows the appearance of voids and cracks in the region close to the failure surface. Helium bubbles, 50 to 150 Å, were seen on ferrite-ferrite grain boundaries and within the ferrite grains (Figure 5b). These grains had a very low dislocation density as contrasted with the austenite grains in which no bubbles were detected.

Samples tested at 800°C did not fail after 300% elongation, the limit of the apparatus. The average grain size increased to between 2 and 4μm and the intergranular cracks were present mostly at ferrite-ferrite boundaries in samples with or without helium as shown in Figure 6. Helium bubbles ranged from 50 to 450 Å in size, and again, were predominantly associated with the ferrite phase.

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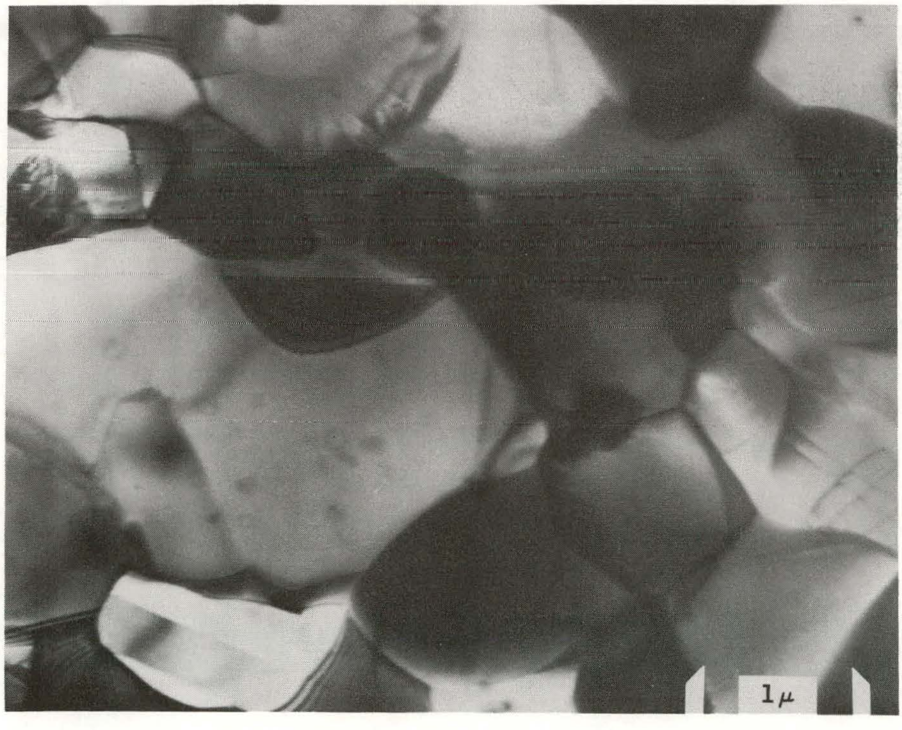
a. Replica-Electron Micrograph



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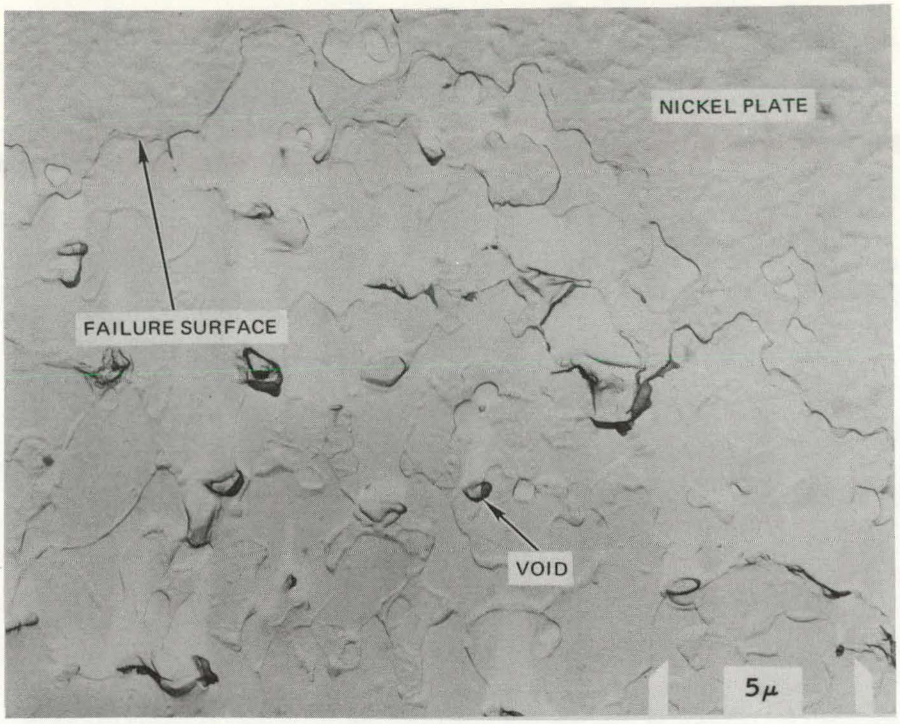
b. Transmission-Electron Micrograph

Figure 4. Microstructure of Inco IN-744X Prior to Testing



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a. Replica-Electron Micrograph Showing Intergranular Voids and Cracks near Failure Surface

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b. Transmission-Electron Micrograph Showing Helium Bubbles in the Ferrite Phase

Figure 5. Microstructure of Inco IN-744X, with 2×10^{-5} Atom Fraction Helium, Tested at 700°C

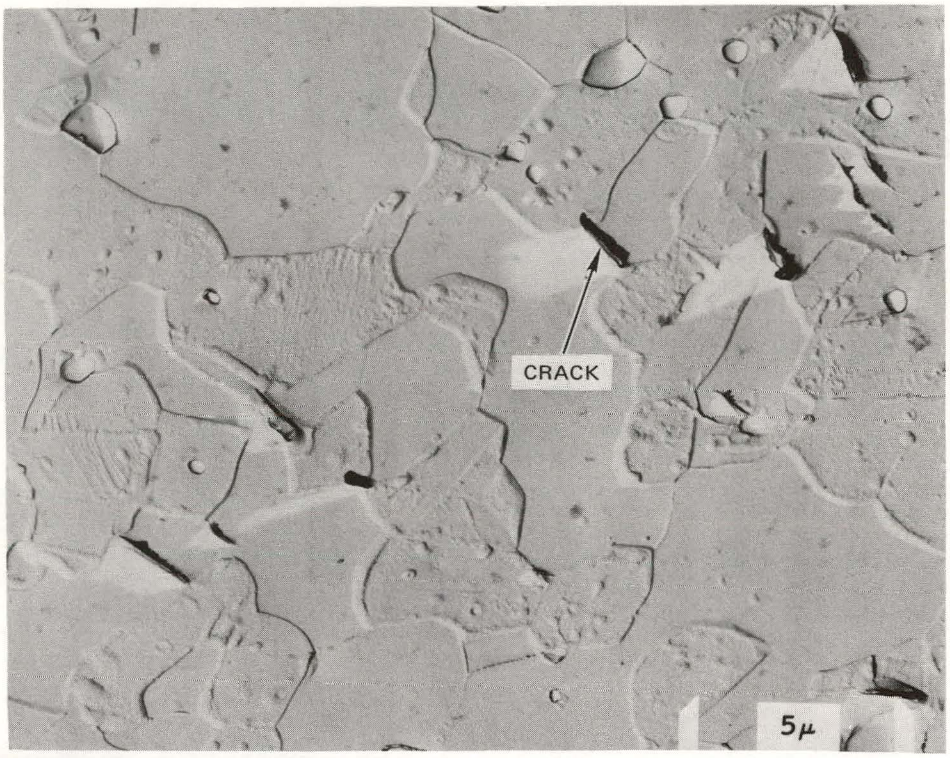

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Figure 6. Replica-Electron Micrograph of Inco IN-744X, Containing No Helium, Elongated 300% Without Failure at 800°C. Cracks are Seen at Ferrite-Ferrite Boundaries.

IV. DISCUSSION

The reduction of total elongation by helium in Sandvik 12R72HV is significantly less than in Types 316 or 304 stainless steel.(5,7) The presence of titanium in Sandvik 12R72HV makes it possible to produce a fine dispersion of $M_{23}C_6$ carbides in the grain boundaries. These precipitates would be expected to impede grain-boundary sliding which is an essential part of the intergranular failure mechanism in Types 316 and 304 stainless steels. In these alloys, intergranular failure was initiated by the formation of voids at grain-boundary carbide particles through the action of grain-boundary sliding; this process is aided by the presence of helium bubbles on the particles. The voids observed in Sandvik 12R72HV were isolated in the grain boundaries, not usually associated with the coarse carbide particles (Figure 2). It is, therefore, likely that grain-boundary sliding was inconsequential and that these voids grew from helium bubbles under the influence of the applied stress.(6) Additional evidence for the reduced grain-boundary sliding in Sandvik 12R72HV as compared to Type 316 stainless steel is given in the Appendix.

The extremely fine grain size and the super-plastic nature of Inco IN-744X put this alloy in a class by itself. Since the mechanism of deformation is uncertain, one is unable to explain the effect of helium in detail. Nevertheless, the extraordinarily high amount of grain-boundary area per unit volume will severely limit locally high concentrations of helium. This is evidenced by the relative scarcity of bubbles seen after testing as compared to Sandvik 12R72HV.

V. CONCLUSIONS

1. Sandvik 12R72HV is significantly more resistant to helium embrittlement than Types 316 or 304 stainless steel.
2. This improved resistance is due to a fine dispersion of $M_{23}C_6$ grain-boundary carbide particles.
3. The presence of helium in Inco IN-744X merely moderates the rapid rise of total elongation with increasing temperature that occurs above 500°C.

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APPENDIX

The expression we have used for elongation due to grain-boundary sliding, ϵ_{gb}, relating the various parameters which can be measured experimentally, as taken from Stevens,$^{(1)}$ is

$$\epsilon_{gb} = (1 + \epsilon_t) \left[1 - (1 + N_T \bar{v}_T)^{-2}\right] \qquad \ldots (1)$$

where

ϵ_t = total elongation

N_T = number of grain boundaries per unit length, transverse to the stress axis, after elongation

$\bar{v}_T$ = average value of grain displacement perpendicular to the stress axis, normal to the surface, transverse traverse.

The equation is valid providing $\theta_1 = \theta_2$, where θ_1 is the angle between the stress axis and the grain-boundary trace on the surface of the sample and θ_2 is the angle between the stress axis and the grain-boundary trace on a section perpendicular to the surface.

Prior to tensile testing 0.025 mm was electropolished from each surface of the sample. This provided a polished surface for displacement measurements and also assured that $\theta_1 = \theta_2$ prior to test (determined experimentally). The grain displacements were measured by means of a Zeiss two-beam interference microscope, with approximately 200 measurements made to determine $\bar{v}_T$. The samples were then sectioned and examined to assure that $\theta_1 = \theta_2$ after test.

The results for Sandvik 12R72HV and Type 316 stainless steel without helium are presented in Table A-1. The samples were tested to their maximum uniform elongations. The parameter γ is $(\epsilon_{gb}/\epsilon_t)$ (100) which is a measure of the relative contribution of grain-boundary sliding to the total elongation. The error limits are the 95% confidence intervals based on the 95% confidence intervals for the $\bar{v}_T$ of each sample.

TABLE A-1

GRAIN-BOUNDARY SLIDING DATA FOR SANDVIK 12R72HV AND TYPE 316 STAINLESS STEEL

Alloy	Test Temperature (°C)	Total Elongation (%)	Elongation Due to G. B. Sliding (%)	γ (%)
12R72HV	800	15	0.62 ± 0.27	4.1 ± 1.8
316 SS	800	9.1	0.89 ± 0.23	9.78 ± 2.5

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1. R. N. Stevens, Grain-Boundary Sliding in Metals, Met. Rev. 11 (1966) 129