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HELIUM EMBRITTLEMENT
OF A
FERRITIC STAINLESS STEEL

AEC Research and Development Report



ATOMICS INTERNATIONAL

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HELIUM EMBRITTLEMENT
OF A
FERRITIC STAINLESS STEEL

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ABSTRACT

Helium was injected into small tensile samples of Type 405 stainless steel by alpha-particle irradiation from a cyclotron. Subsequent tensile testing at 550, 650, and 750°C revealed an increasing loss of ductility with temperature, which reached 55% at 750°C.

The presence of helium caused the failure mode to change from trans- to intergranular failure. The grain-boundary cracks originated as small voids adjacent to grain-boundary carbide particles. Large helium bubbles, found attached to carbide particles, are responsible for the void formation, presumably through the action of grain-boundary sliding.

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I. INTRODUCTION.

Exposure of steels to a fast-neutron flux results in helium generation by (n, α) reactions. The elements Fe, Ni, Cr, and N are major participants in the (n, α) reaction, and helium concentrations of up to 5×10^{-5} atom fraction could accrue in fuel claddings after a year of service in proposed fast reactors.⁽¹⁾

Such concentrations of helium can severely reduce the ductility of alloys at temperatures of reactor service. The phenomenon of helium embrittlement has been studied in austenitic alloys which were injected with α particles by cyclotron irradiation, as a rapid means of producing significant helium concentrations.⁽²⁻⁴⁾ The present work is a continuation of those studies, and is concerned with helium effects in a ferritic stainless steel.

II. EXPERIMENT

Small sheet tensile samples, with a gauge length of 0.50 in. and a cross section of 0.009 in. by 0.040 in. in the gauge length, were punched from cold-rolled Type 405 stainless steel. The composition of the steel used in this work is given in Table 1.

TABLE 1
COMPOSITION OF TYPE 405 STAINLESS STEEL

(wt %)						
Cr	C	Ni	Si	Mn	Al	Fe
15.4	0.056	0.27	0.39	0.56	0.15	bal.

Prior to helium injection, the samples were annealed for 24 hr at 760°C, which caused complete recrystallization with a resulting grain size of 40 μ . This treatment renders the alloy completely ferritic; and carbide particles, 0.5 to 2 μ in size, are present at a density of $1 \times 10^{11} \text{ cm}^{-3}$. These carbide particles are probably a mixture of the M_7C_3 and M_4C types.⁽⁵⁾

Helium was injected into the samples, by irradiation with α particles from a cyclotron, to concentrations of 2.5 and 4.0×10^{-5} atom fraction. Details of this procedure were described previously.⁽²⁾ A gas-source mass spectrometer was employed to measure the helium concentration in the samples.

After helium injection, samples were tensile tested in vacuum at 550, 650, and 750°C, using a strain rate of 0.02 min.⁻¹. Metallography was used to ascertain the nature of the tensile failure and to understand the mechanism of the helium effect. Additional experiments were performed to learn more about the interaction of helium with dislocations.

III. RESULTS

A. TENSILE TESTS

The results of the tensile tests are given in Table 2, where the values shown are averages of two or more samples. There was no significant effect of helium on either the yield strength or tensile strength. Displacement damage caused by the α irradiation anneals out rapidly, at temperatures of interest here. Samples with helium showed a 20% loss in total elongation at 650°C, which increased to a 55% loss at 750°C.

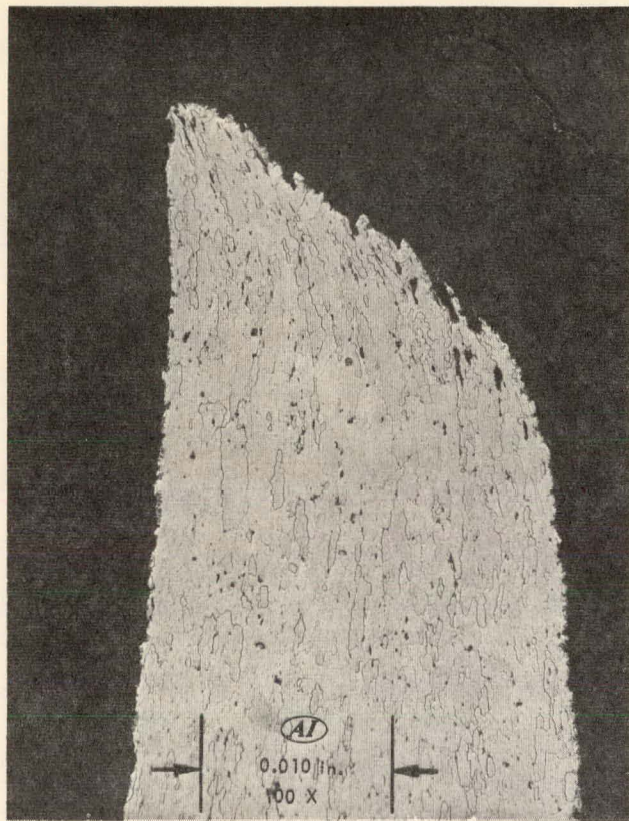
TABLE 2
TENSILE DATA OF TYPE 405 STAINLESS STEEL SAMPLES

Test Temperature (°C)	Atom Fraction Helium	Yield Strength (kpsi)	Tensile Strength (kpsi)	Elongation (%)	
				Uniform	Total
550	0	16.8	26.3	12	46
550	4.0×10^{-5}	15.4	26.3	10	44
650	0	9.8	12.1	6.4	54
650	4.0×10^{-5}	10.7	12.7	7.0	43
750	0	4.5	4.7	1.7	49
750	4.0×10^{-5}	5.8	6.3	3.5	22

B. METALLOGRAPHY OF SAMPLES TESTED AT 750°C

Photomicrographs near the fracture edge of samples without helium and with helium are shown in Figure 1. A few voids and cracks are visible in the sample without helium, and these are located both in grains and on grain boundaries. The sample containing helium has extensive intergranular cracks in the fracture zone.

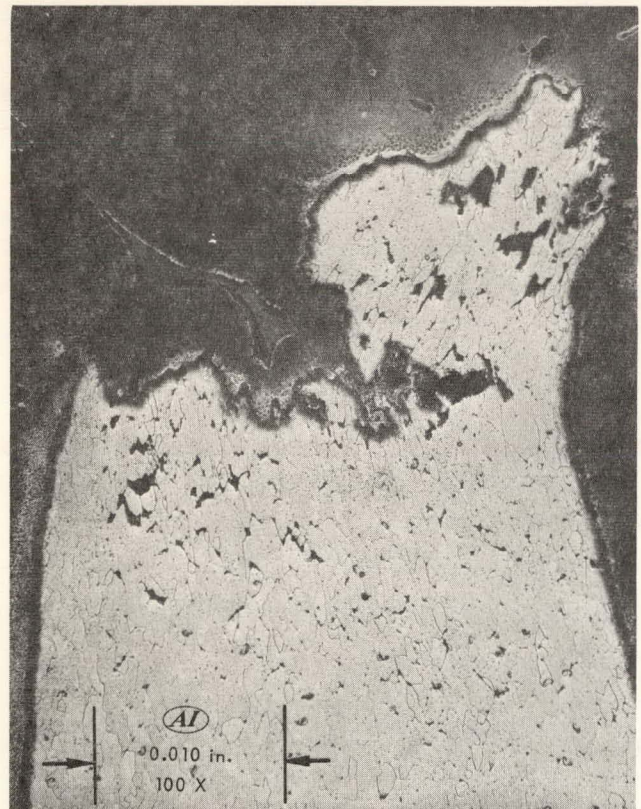
The appearance of the samples in Figure 1 indicated that samples without helium failed transgranularly, whereas the presence of helium caused the samples to fail intergranularly. The natures of the failures were confirmed by fractography, which revealed typical shear-rupture dimples in the absence of helium and grain boundaries separated by relatively smooth areas in the presence of helium.



a. Without Helium

7706-4770

b. With 4.0×10^{-5} Atom
Fraction Helium



7706-4771

Figure 1. Photomicrographs of Type 405 Stainless Steel Samples
Tensile Tested at 750°C

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Replicas for electron microscopy were taken of samples on a plane parallel to the tensile axis and to their broad surface, to detect incipient cracks and voids. These negative replicas reveal a depression on the sample surface by the presence of a light shadow adjacent to a dark area.

Figures 2 and 3 are replica electron micrographs of samples without helium and with helium respectively. Referring to both figures, voids are seen adjacent to matrix carbide particles at A and adjacent to a grain-boundary carbide at B. A grain boundary crack is present at C, and large voids are located at D. Carbide particles were favored sites for void formation, either within grains or on grain boundaries, as determined from observations of many replicas. Considerable polygonization was observed in grains near the failure region.

Transmission electron micrographs on samples without helium (Figure 4) and with helium (Figures 5 and 6) show a low dislocation density. The grain boundary in Figure 4 was in the process of migration, and was being impeded by several carbide particles, which resulted in cusps. This evidence for grain-boundary migration was common in samples without and with helium.

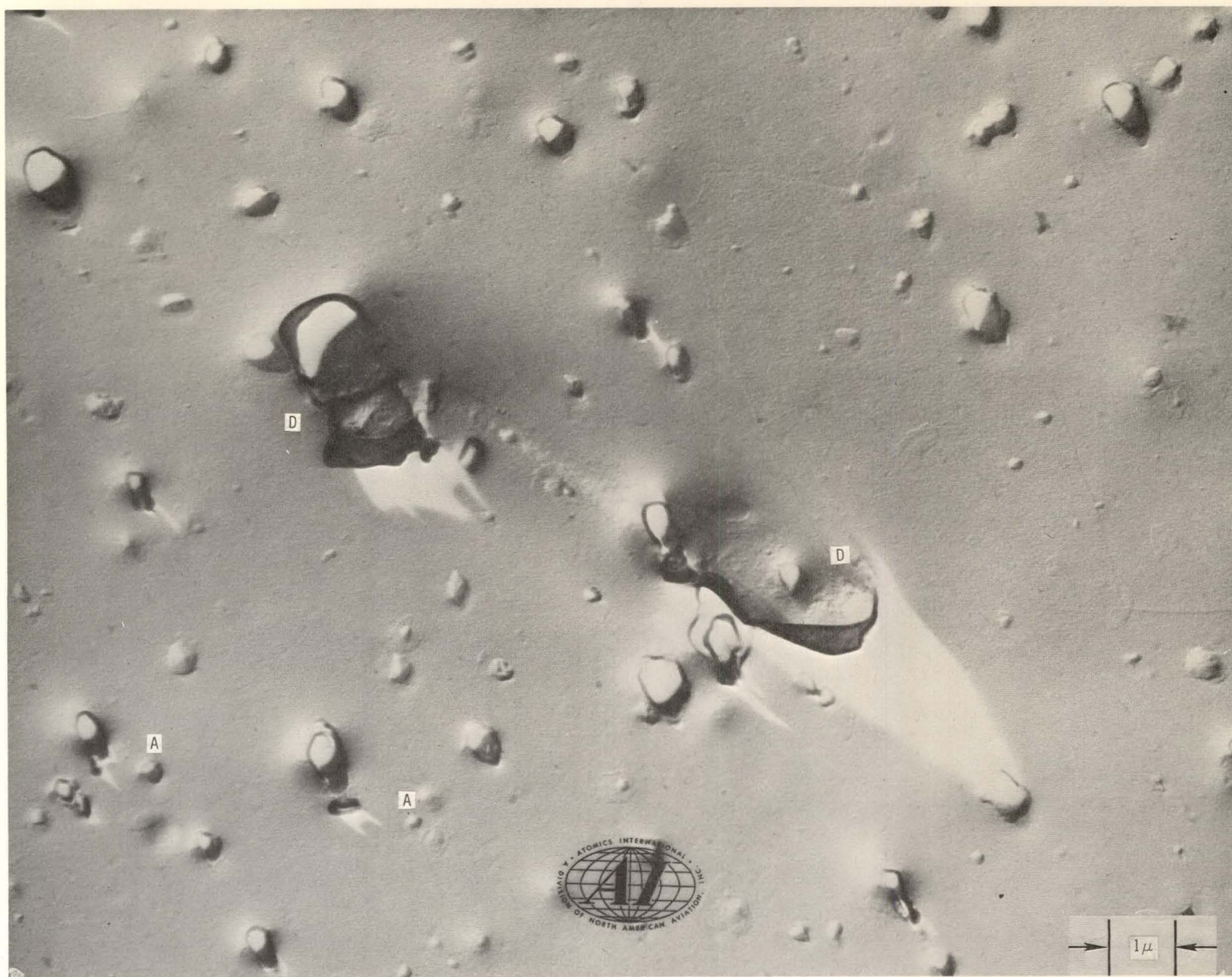
Large helium bubbles, greater than 1000\AA , were found on some grain boundaries and on carbide particles located on grain boundaries, as seen in Figures 5 and 6. Smaller bubbles, 100 and 500\AA , were observed in the matrix, on matrix carbide particles, and on other grain boundaries.

C. EFFECT OF STRAIN AND TEMPERATURE ON BUBBLE FORMATION

The results of several combinations of strain and temperature on the formation of observable bubbles are given in Table 3. Such bubbles were only seen when the samples were strained at 750°C . Merely annealing at 750°C , or straining at room temperature followed by annealing at 750°C , did not produce bubbles.

In all cases, the samples were strained to failure. It is noteworthy that the helium-containing sample tested at room temperature retained a high dislocation density after annealing at 750°C .

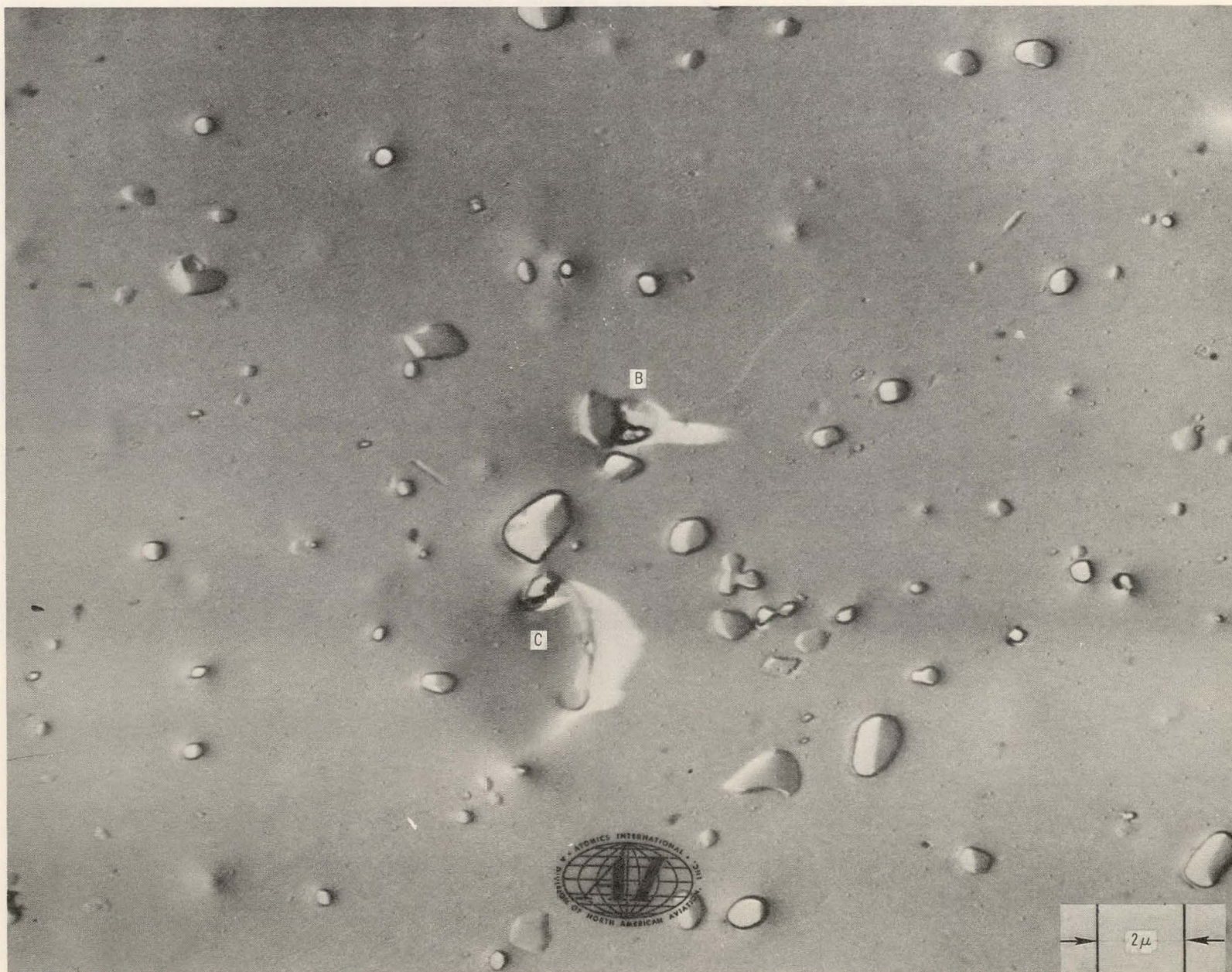
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Figure 2. Replica Electron Micrograph of Type 405 Stainless Steel, Without Helium, Tested at 750°C, Showing Voids at Carbide Particles

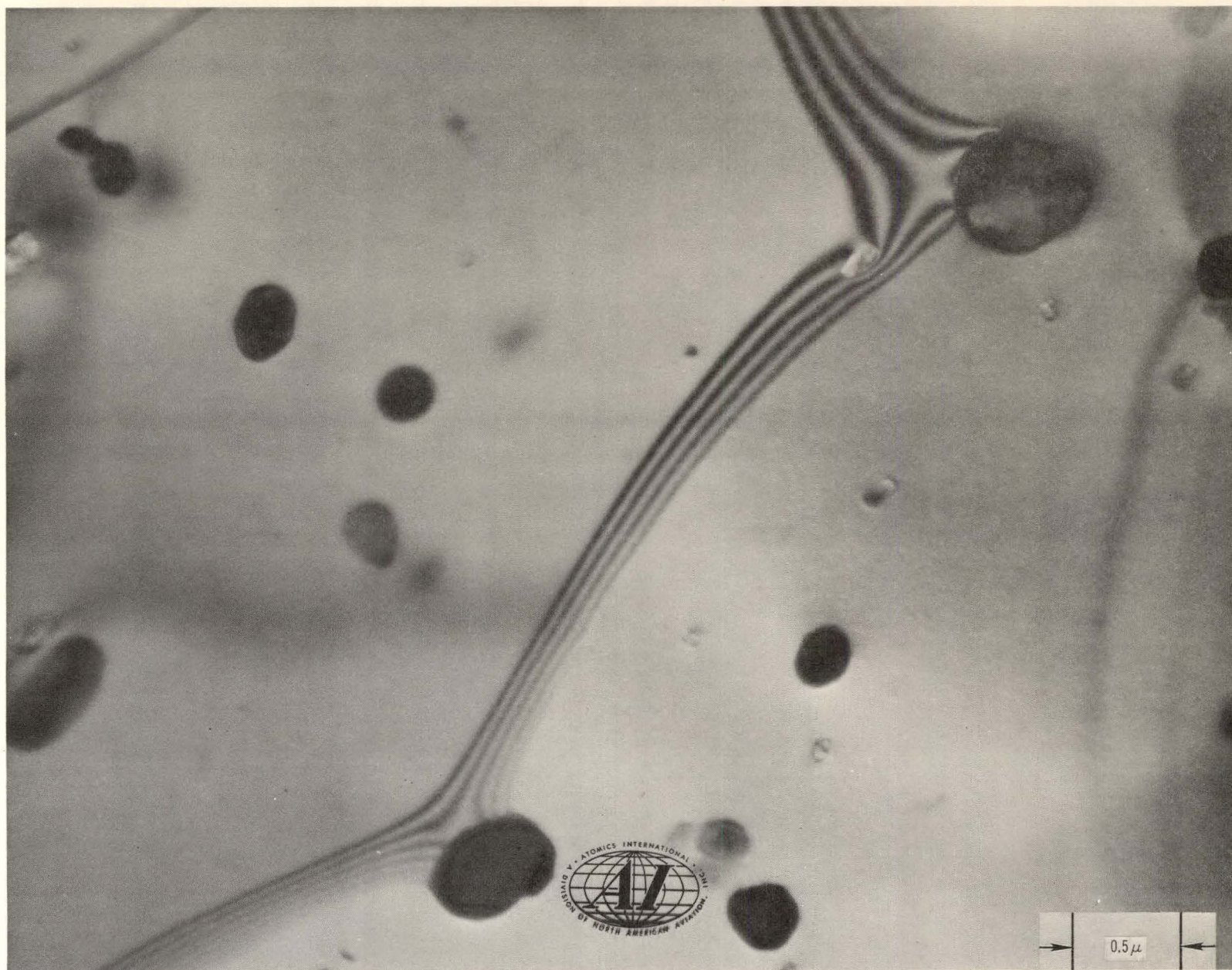
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Figure 3. Replica Electron Micrograph of Type 405 Stainless Steel, With 4.0×10^{-5} Atom Fraction Helium, Tested at 750°C , Showing Voids at Carbide Particles and Grain-Boundary Crack

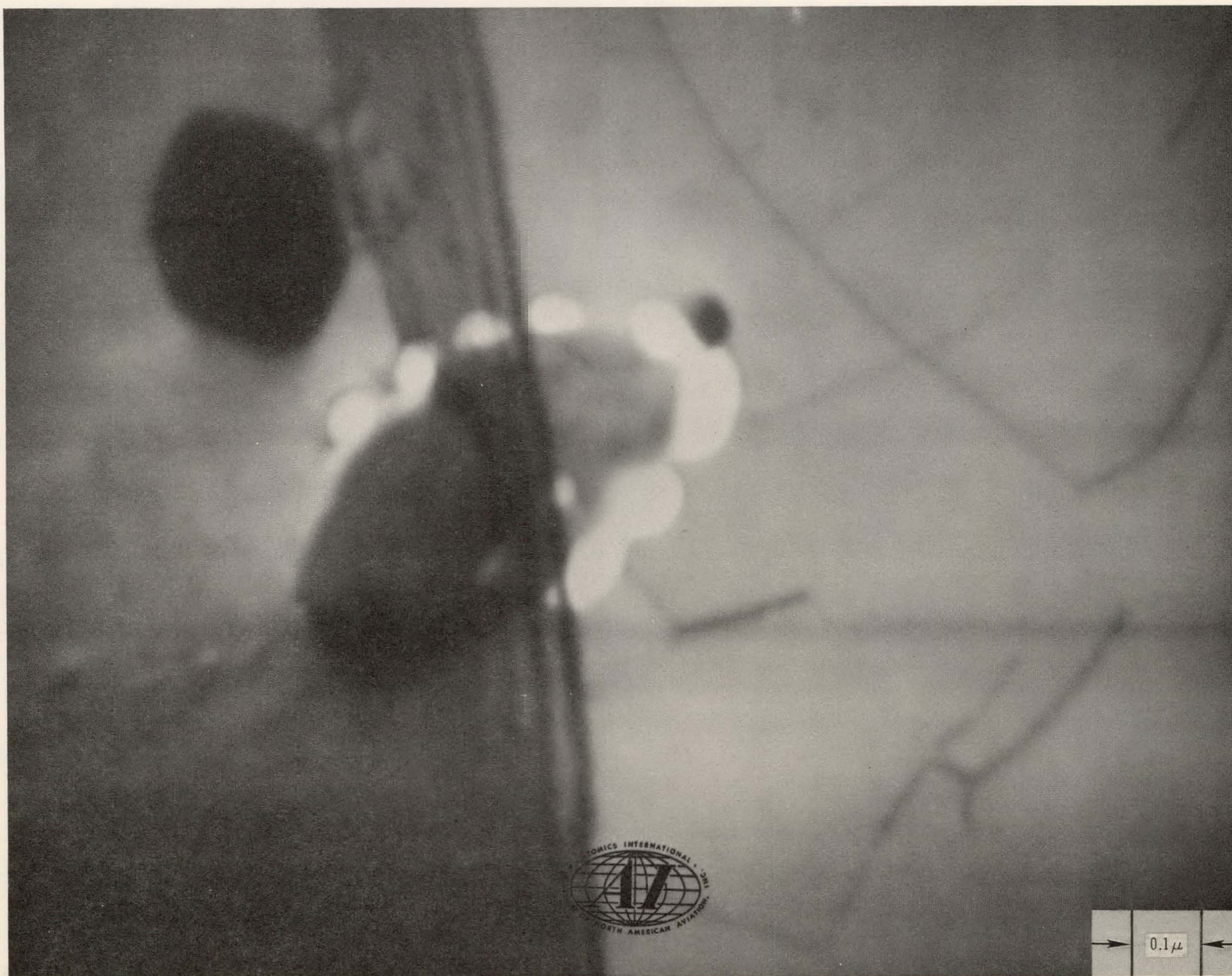
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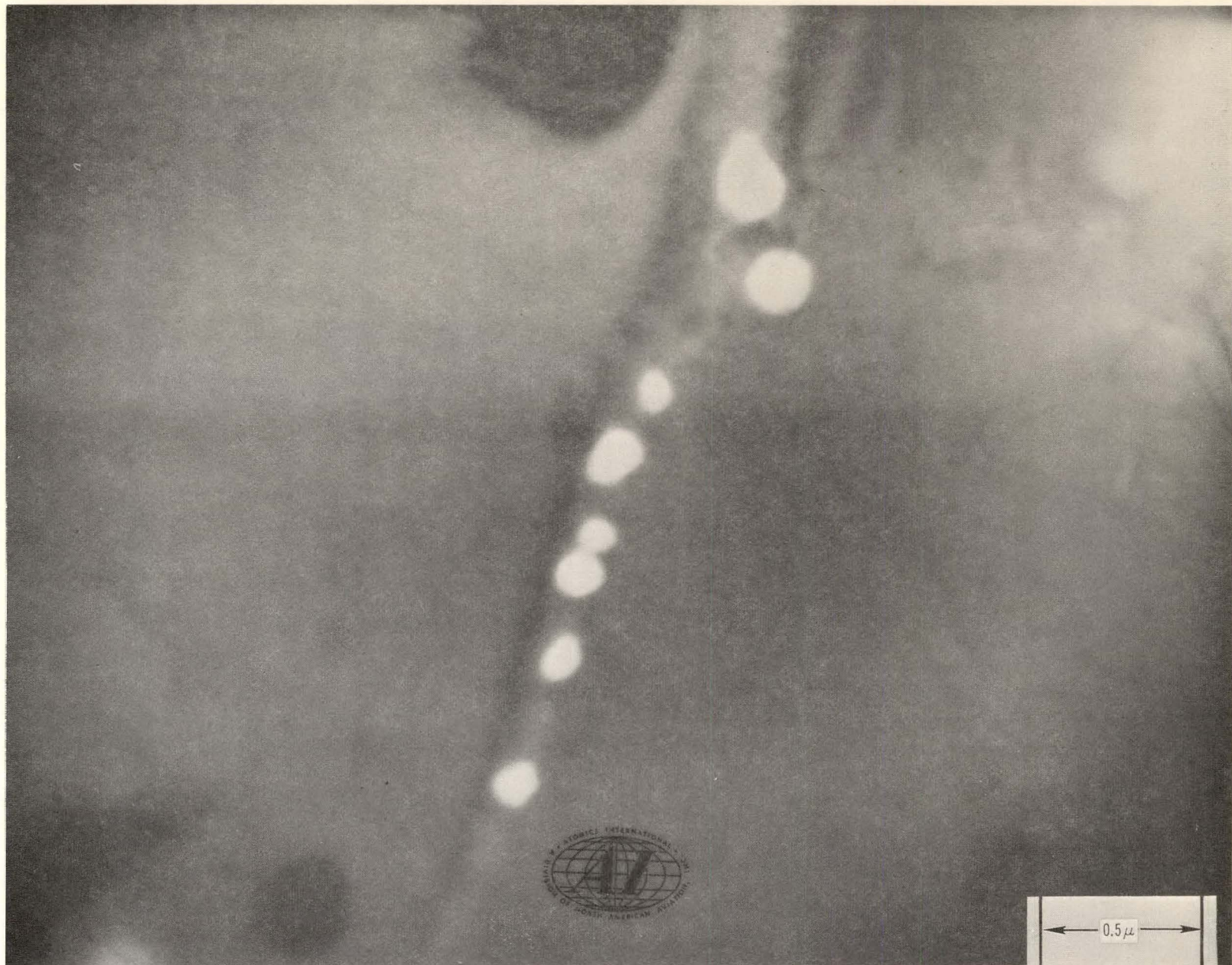
Figure 4. Transmission Electron Micrograph of Type 405 Stainless Steel, Without Helium, Tested at 750°C, Showing Migrating Grain Boundary

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Figure 5. Transmission Electron Micrograph of Type 405 Stainless Steel, With 4.0×10^{-5} Atom Fraction Helium, Tested at 750°C, Showing Helium Bubbles on Grain-Boundary Carbide Particle



22-107

Figure 6. Transmission Electron Micrograph of Type 405 Stainless Steel, With 4.0×10^{-5} Atom Fraction Helium, Tested at 750°C , Showing Helium Bubbles on Grain Boundary

TABLE 3
EFFECT OF STRAIN AND TEMPERATURE
ON BUBBLE FORMATION

Atom Fraction Helium	Test Temperature (°C)	Anneal at 750° C (hr)	Dislocation Content	Helium Bubbles
0	750	None	Low	-
2.5×10^{-5}	750	None	Low	Yes
2.5×10^{-5}	Untested	2	Very low	No
0	Room	2	Low	-
2.5×10^{-5}	Room	2	High	No

IV. DISCUSSION

The general tensile behavior of Type 405 stainless steel containing helium is similar to that of other austenitic alloys previously studied.⁽²⁻⁴⁾ The ductility loss between 600 and 800°C is less for Type 405 stainless steel than was found for either Type 316 stainless steel or Incoloy 800 with the same helium contents and with equivalent microstructures.

The mechanism of helium embrittlement is also the same, and has been previously discussed in more detail.^(2,3) Briefly, the presence of helium caused the failure to change from transgranular to intergranular at 750°C. The replica electron micrographs showed that the grain-boundary cracks originated as small voids, adjacent to carbide particles located on the boundary. Large amounts of helium can collect on these carbides (Figure 5), making them likely sites for crack nucleation under the action of grain-boundary sliding.

The reduced susceptibility of Type 405 stainless steel to helium embrittlement may be due to the relative ease of grain-boundary migration, as compared to the austenitic stainless steels. The buildup of high shear stresses at grain-boundary carbides, due to grain-boundary sliding, will be retarded if the grain boundary is moving.

The results shown in Table 3 indicate that strain at high temperature is effective in forming helium bubbles. This is in agreement with Barnes' theory on the role of moving dislocations in promoting bubble growth by a sweeping action.⁽⁶⁾ Bubbles will form in the absence of strain, but at higher temperatures and longer times.

The retention of a high dislocation density in the helium-containing sample tested at room temperature and annealed at 750°C means that the dislocations were pinned by unresolvable helium bubbles or clusters attached to them. Such small clusters were probably also formed by a dislocation sweeping mechanism.

V. CONCLUSIONS

- 1) Helium at a concentration of 4.0×10^{-5} atom fraction causes a ductility loss during tensile testing between 550 and 750°C.
- 2) Loss of ductility is associated with intergranular cracking, which originates at grain-boundary carbide particles.
- 3) Large helium bubbles attached to grain-boundary carbide particles are responsible for the initiation of cracks, presumably through the action of grain-boundary sliding.
- 4) The behavior of ferritic Type 405 stainless steel containing helium is similar to that of the austenitic stainless steels.

REFERENCES

1. A. DePino, J. Nucl. Appl., 3 (1967) p 620
2. D. Kramer, H. R. Brager, C. G. Rhodes, and A. G. Pard, J. Nuc. Mat., 25 (1968) p 121
3. D. Kramer, K. R. Garr, C. G. Rhodes, and A. G. Pard, J.I.S.I. (to be published)
4. K. R. Garr, D. Kramer, C. G. Rhodes, and A. G. Pard, J. Nuc. Mat. (to be published)
5. M. G. Gemmill. The Technology and Properties of Ferrous Alloys for High-Temperature Use (George Newnes Ltd., Dorking, England, 1966)
6. R. S. Barnes, Flow and Fracture of Metals and Alloys in Nuclear Environments (ASTM, Philadelphia, 1965) p 40

