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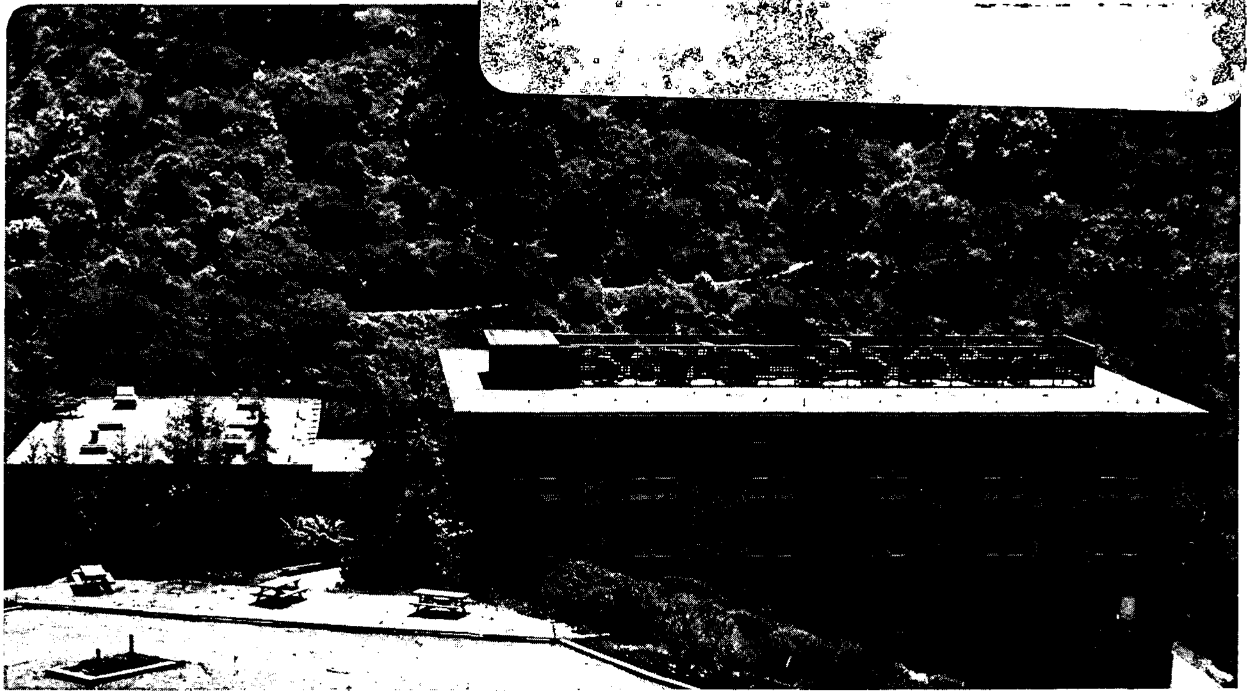

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Key words: cryogenics, mechanical properties, MP35N

INTRODUCTION

MP35N was developed by the E. I. DuPont de Nemours Company for service as a corrosion-resistant alloy with extremely high strength [1]. The chemical composition of MP35N is approximately 35% Ni, 35% Co, 20% Cr, and 10% Mo. The material assumes the fcc crystal structure at temperatures above 650°C, but is metastable with respect to a fcc→hcp phase transformation at lower temperatures. This phase transformation is apparently an athermal martensitic transformation that can be promoted by plastic deformation. Like the fcc→bcc martensite transformation of 304 stainless steel, the phase transformation in MP35N serves as a potent strengthening mechanism. Additionally, the formation of Co_3Mo precipitates during aging at 550°C is a second strengthening mechanism for MP35N.

EXPERIMENTAL METHODS

The material for our mechanical tests was induction melted and vacuum arc remelted by the Latrobe Steel Company. Specimens were cut from stock with about 45 % prior cold work, but were unaged. Round tensile specimens of 3.2 mm gage diameter and 1.8 cm gage length were machined with their tensile axes parallel to the rolling direction. The compact specimen for toughness testing received a final grinding to 1.27 cm thickness. It had a crack plane orientation T-L, and a 3 mm precrack was developed at 77 K. All mechanical tests employed the cryogenic mechanical properties test facility described previously [2]. A strain rate of 10^{-4} /sec was used for all tensile tests, and a crosshead velocity of 2.5×10^{-4} cm/sec was used for the toughness test. Stress relaxation tests at 4 K and 77 K were performed by stopping specimen elongation in strain control mode and recording any reductions in applied load. Temperatures of 4.2 K and 77 K were obtained with the specimens submerged in liquid helium and liquid nitrogen, respectively. Three tensile specimens were tested at 77 K and two at 4 K. One compact toughness specimen was tested at 4 K. An ISI scanning electron microscope operated at 20 kV was used for a fractographic examination of the toughness specimen.

RESULTS AND DISCUSSION

Stress-strain curves from the tensile tests at 4 K and 77 K are shown in fig 1. Our measured Young's modulus is lower than that reported by Montano [3]. A gradual decrease in the slope of the stress-strain curve before yielding was observed, especially at 4 K. At 4 K the 0.2 % offset yield strength of 1.7 GPa does not give a full indication of the strength of MP35N. When a material exhibits a gradual departure from elastic behavior, a small change in the offset criterion may cause a large change in the determination of yield strength. Using an offset criterion of less than 1 %, the 4 K yield strength is over 2 GPa, and this may be a more appropriate measure of the strength in some applications. The tensile behavior of MP35N is unique in its remarkably gradual rate of work-hardening after about 2 % plastic strain. By the Consideré criterion, a constant force during tensile deformation represents the minimum amount of work-hardening that can suppress immediate necking. It may therefore be expected that small material variations that cause small variations in work-hardening behavior could have major effects on the uniform elongation. Nevertheless, only small differences in the total elongations of the tensile specimens were observed. Plastic flow at 4 K showed the jerky behavior that is a common characteristic of tensile behavior below 20 K. It is interesting to note that the flow strength at 4 K is not significantly greater than the flow strength at 77 K, and during load drops the stress fell below the flow stress at 77 K. These observations are inconsistent with the Basinski mechanism for jerky flow [4] unless local temperature excursions in the specimen exceeded 77 K during the load drops.

TABLE I. MP35N CRYOGENIC TENSILE PROPERTIES
(45 % Cold Work , Unaged)

Temp.	Modulus	0.2% Yield	Ultimate	Red. Area	Tot. Elong.
77 K	1.6×10^{11} Pa	1.93 GPa	2.41 GPa	42 %	17 %
4 K	1.8×10^{11} Pa	1.7 GPa	2.55 GPa	30 %	14 %

Measurements of the path-invariant elastic-plastic fracture toughness, J_{IC}, were performed according to the procedure proposed by Dalder [5], which uses the unloading compliance of the specimen to determine the instantaneous crack length. The measured force versus crack-opening-displacement curve is shown in fig 2. The elastic crack opening ended abruptly when a "pop" was clearly audible and the load on the specimen decreased rapidly. With further increase in crack opening displacement the load would rise slowly and drop suddenly as the crack "popped-in". The early pop-in allowed the determination of a valid K_{IC} in accordance with ASTM Standard E-399 [4]. For our specimen the load of the first pop-in corresponded to a K_{IC} of 102 $\mathrm{MPa \cdot m^{1/2}}$. This is a valid K_{IC} because the 0.5 inch specimen thickness is much greater than the square of the ratio of K_{IC} to σ_y, where σ_y is the yield strength. The 95 % slope rule of ASTM Standard E-399 was satisfied by this test. Although 102 $\mathrm{MPa \cdot m^{1/2}}$ would be a respectable fracture toughness for many materials of lower strength, the important criterion for determining notch sensitivity is the critical flaw size, ρ:

$$\rho = f \cdot (K_{IC}/\sigma_y)^2 \quad .$$

Here f is a geometrical factor unique to each type of crack. In engineering practice a significant safety factor is also included in the factor f. If $f = 1$, then the critical flaw size for MP35N at 4 K is about 2.4 mm. A more practical value of f will probably place the critical flaw size around ½ mm - 1 mm. Unfortunately, the detection of a crack of this size could be difficult. It should be pointed out that crack propagation along the rolling direction usually represents the easiest direction for crack propagation in a metal. Therefore the K_{IC} determined from the present test with T-L crack plane orientation probably represents a lower bound for the useful 4 K fracture toughness of MP35N. Nevertheless, the combination of strength and toughness of MP35N at 4 K lies well beyond the trend line for 304 stainless steel [7].

Our data show that cold-worked MP35N exhibits a departure from simple elastic behavior at stresses below the conventional 0.2 % offset yield strength. Similar behavior is well-documented in 304L stainless steel, which undergoes a martensitic transformation when under stress [2,8]. We suspect that this similar behavior of MP35N is a consequence of some fcc→hcp phase transformation that occurs during loading. Immediately prior to a tensile test at 77 K and 4 K, the specimen was loaded to half of its yield stress and then unloaded. The curvature of the stress-strain curve below the yield stress was especially evident above 1.5 GPa. This curvature was not observed during unloading. During subsequent loading the stress-strain curve was a straight line up to the maximum load of the first loading, but again rolled off at higher loads. These observations of the load, unload, reload cycle suggest that the fcc→hcp phase transformation was effectively completed for the stresses developed by the first loading. However, the phase transformation began again when the stress rose above that of the first loading.

These latter observations suggest a way to achieve higher 0.2 % offset yield strengths in MP35N components subject to tensile loading. The data used for fig 3 show that a few percent plastic prestrain at 4 K allows the 0.2 % offset yield strength to exceed 2 GPa upon the second loading. We suggest that a plastic prestrain of 2 or 3 % at the service temperature should be sufficient to maximize the yield strength. A second use for plastic prestrain at low temperatures could be as a test for critical flaws. Components that survive this deformation without fracture will have passed a practical inspection for critical flaws. We believe that the scanning electron fractographic examination suggests the prudence of such a proof test. The micrographs of fig 4 show an inhomogeneous mixture of both a ductile-dimple fracture mode and what appears to be a more brittle quasi-cleavage type of fracture mode. An internal crack perpendicular to the fracture surface is seen in fig 4b. We currently have too little data to know whether the amount of quasi-cleavage fracture or the number of internal cracks will vary from heat to heat of material.

Large cryogenic creep rates have been reported previously for some stainless steels which are metastable with respect to the martensite transformation [2,9]. By analogy, the fcc→hcp phase transformation in MP35N could also be associated with cryogenic creep. No detectable load relaxation of a MP35N tensile specimen at 4 K was found during three 5 min holdings after both elastic and plastic strains. No detectable load relaxations were found at 77 K during 20 minute holdings at a stress of 1.3 GPa and after 3% plastic strain. This dimensional stability is encouraging, but effects over longer times are yet unexplored.

CONCLUSIONS

The alloy MP35N in the unaged, 45 % cold-worked condition has excellent strength at 77 K and 4 K, together with reasonable ductility in a tensile test. For a given uniform elongation and reduction in area, its ultimate tensile strength is greater at cryogenic temperatures than at room temperature. The 0.2 % yield strength of this material can be significantly improved by a small amount of plastic deformation at low temperatures. Short time tests have shown no creep in this material at 4 K. MP35N is, however, somewhat notch-sensitive.

ACKNOWLEDGMENTS

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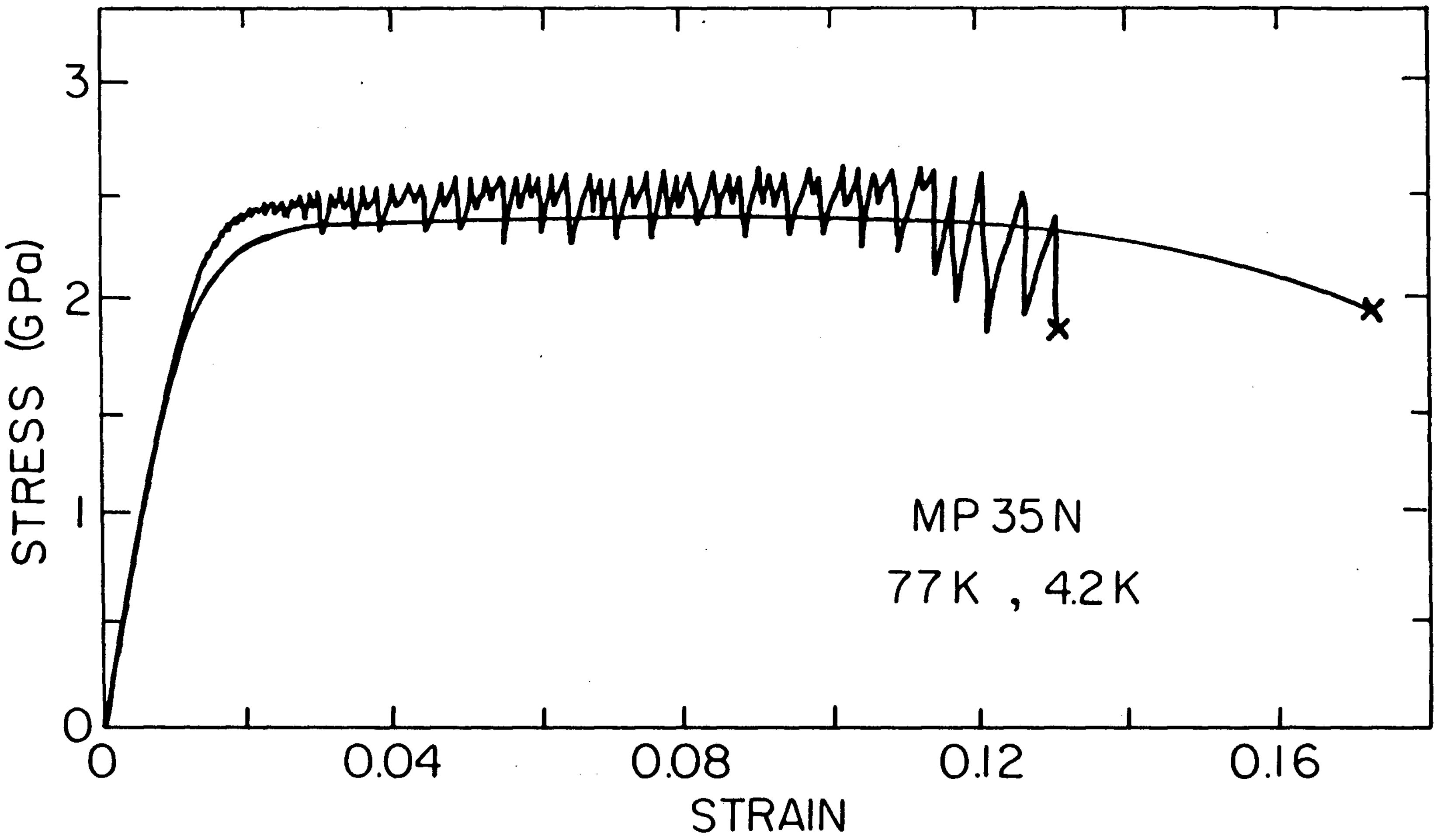

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Fig. 1 Engineering stress-strain curve of 45 % cold-worked and unaged MP35N at 77 K (smooth curve) and at 4 K (with serrations).

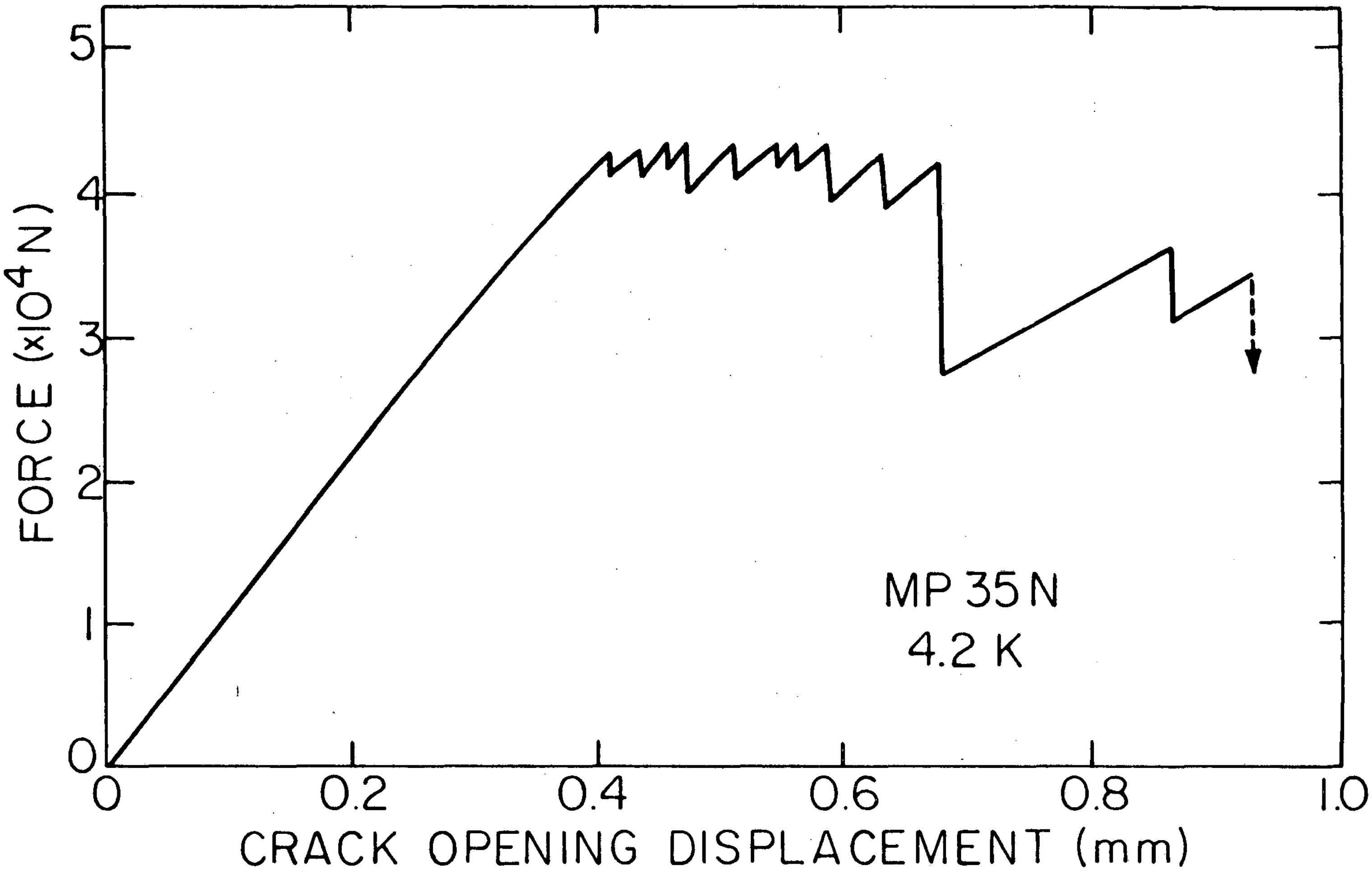

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Fig. 2 Load versus crack opening displacement from J_{Ic} test of cold-worked and unaged MP35N at 4 K.

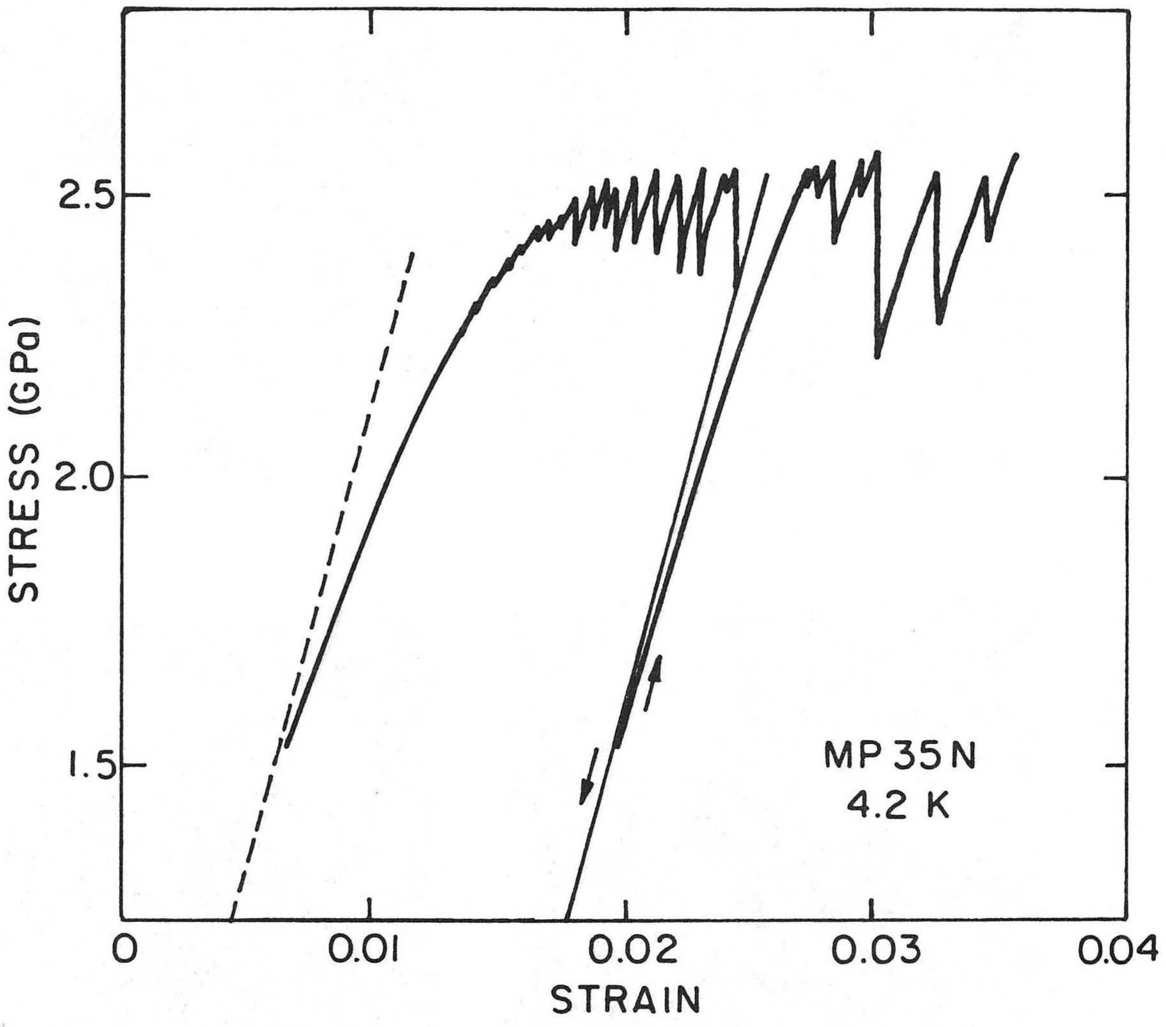

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Fig. 3 Load/unload stress-strain curves of 45 % cold-worked and unaged MP35N at 4 K. a- unloading after elastic strain at 77 K, b- unloading after plastic strain at 4 K

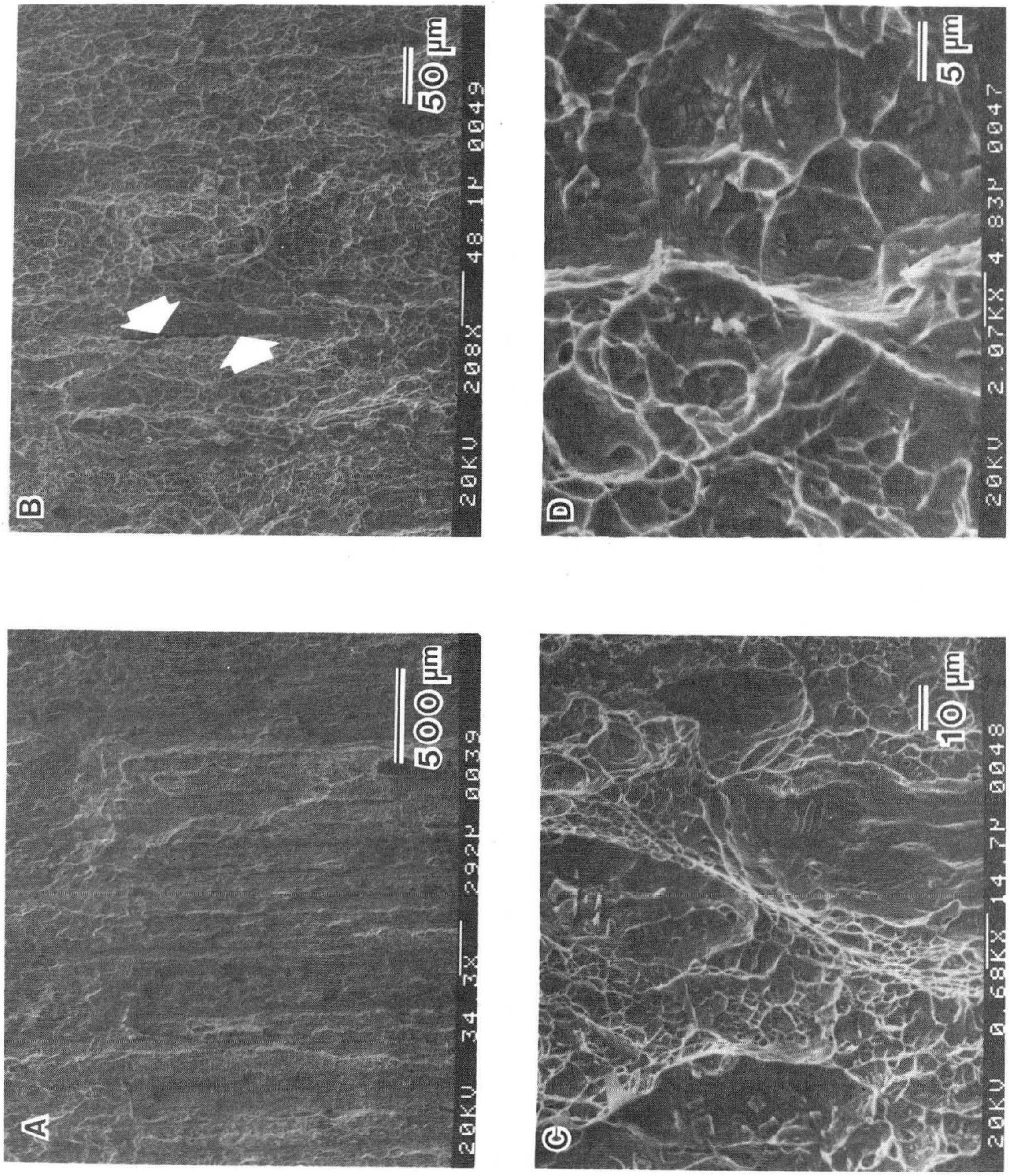

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Figure 4

Fig. 4 Scanning electron micrographs from the fracture surface of the MP35N compact toughness specimen. a- very low magnification overview of surface, b- low magnification image showing internal crack, c- medium magnification image showing mixed-mode fracture, d- high magnification image of dimpled region showing particles in the dimples

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