## Title

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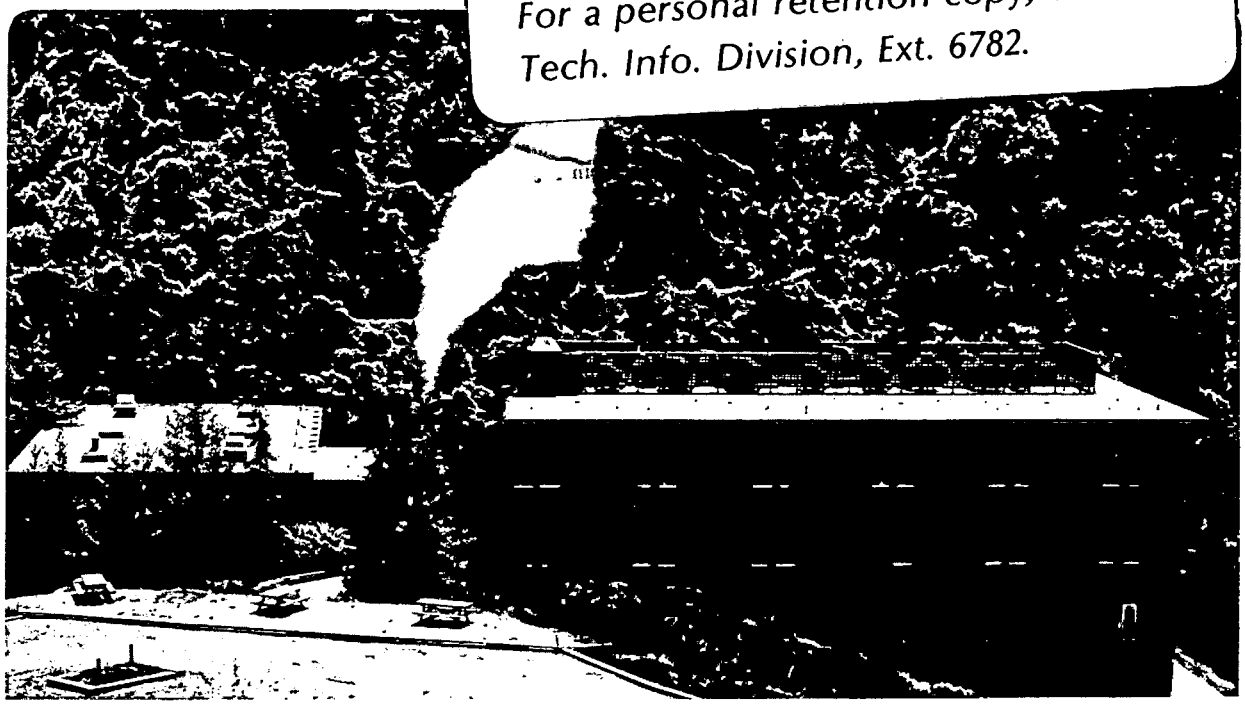

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# The Effect of V and V+Ti on the Magnetic and Mechanical Properties of Fe-Cr-Co Hard Magnets

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## ABSTRACT

The microstructure and magnetic properties of the Fe-Cr-Co hard permanent magnetic alloys with and without V and V+Ti are studied using transmission electron microscopy and transmission Lorentz microscopy. Mechanical and magnetic properties are studied in parallel at different stages of their production. The three alloys 57Fe-15Co-28(Cr,V, Ti) used for the present investigation have 28Cr, 23Cr-5V and 23Cr-3V-2Ti, respectively. It is known that the magnetic properties of these alloys are improved by a heat treatment in a magnetic field. Improvement in magnetic remanence, $B_r$ is due to the elongation of Fe-Co ($\alpha_1$) phase parallel to the magnetic field direction. Subsequent step aging or continuous cooling treatments cause further phase separation with increased compositional differences between the phases without any microstructural changes and thus increase the coercive field $H_c$. Lorentz microscopy shows that the matrix is weakly magnetic and the magnetic hardening is due to domain wall pinning. Mechanical testing and fracture analysis show that the alloys are very ductile in the as-quenched state, but become very brittle after the aging treatments. The embrittlement is very severe for the ternary alloys and is most effectively

retarded by the addition of V or V+Ti. This is attributed to the high Cr content of the Cr-rich ($\alpha_2$) phase and to the large grain size (a result of high homogenization temperature) of the ternary alloys. Since the addition of V or V+Ti does not change the magnetic properties, but improves the mechanical properties and makes the processing more convenient, the alloys with V and V+Ti seem to be of commercial interest.

## I. Introduction

Fe-Cr-Co alloys with magnetic properties comparable to those of alnico permanent magnets have been developed in recent years[1,2,3]. The advantage of the Fe-Cr-Co alloys lies in their useful mechanical properties such as ductility and fracture toughness. However, these alloys are ductile only in the solution treated stage and lose their ductility after they are optimally aged for good magnetic properties.[3] This paper presents the results of our investigation to design Fe-Cr-Co alloys combining the best magnetic properties with the best mechanical properties through an understanding of their dependences on the microstructure.

## II. Experimental Procedure

The alloy compositions used in the present investigation are: Fe-28Cr-15Co, Fe-23Cr-15Co-5V, Fe-23Cr-15Co-3V-2Ti (wt.%), designated as alloys A, B, and C, respectively. Alloy A was homogenized at 1300°C and alloys B and C could be homogenized at 1000°C due to the presence of V and V+Ti. After homogenizing for one hour in an argon atmosphere, the alloys were quenched in ice water and aged at lower temperatures (details of aging appear in the next section). Magnetic properties were measured using 5mm diameter and 30mm long bars with an automatic recording flux meter. Tensile specimens with a 0.318 cm gauge diameter and 1.91 cm gauge length were pulled to fracture using an Instron testing machine. Fracture surfaces were examined with an AMR scanning electron microscope. Philips EM301 and JEOL JEM7 electron microscopes were used in the transmission electron microscopy and Lorentz microscopy studies.

## III. Results and Discussion

### A. Microstructure-Magnetic Property Relationships:

(i) Isothermal aging: Fig. 1 shows the bright field (B.F.)

micrographs taken from Alloy A after aging at 670°C, 650°C and 640°C, respectively for one hour. The phase with bright contrast is identified as $\alpha_1$ (Fe-rich phase) and the one with dark contrast as the $\alpha_2$ (Cr-rich) phase. From these micrographs, it is seen that the morphology of the microstructure, i.e., the shape, size and volume fractions of the two phases is very sensitive to the aging temperature. This is due to the asymmetry in the miscibility gap in the phase diagram.[4]

(ii) Thermomagnetic treatment (TMT) and Step Aging: It is known that TMT is important for producing optimum magnetic properties.[5] Fig. 2 shows the BF micrographs of Alloy B aged at 650°C for one hour without and with a magnetic field. The effect of TMT on the microstructure is seen clearly in Fig. 2(b) in that the $\alpha_1$ phase is remarkably elongated along the direction of the applied field, whereas in Fig. 2(a) the elongation of the $\alpha_1$ phase is negligible and the orientation of the particles is seen to be almost random.

It is known that optimum magnetic properties can be produced by step aging or continuous cooling of the thermomagnetically treated alloys.[6] When these alloys are step aged at lower temperatures, the composition difference between the two phases increases without affecting the essential features of the morphology. The effect of step aging and also the effect of changing the temperature of TMT on the microstructure and the properties were studied in detail. Figure 3 shows BF micrographs taken from Alloy A after TMT and step aging. The TMT temperatures were a) 670°C, b) 660°C, c) 650°C and d) 640°C, respectively. All the alloys were step aged at 620°, 600°, 580°, and 560°C for one hour each and at 540°C for five hours. Figures 3(a) and (b) show secondary decomposition of $\alpha_2$ phase during step aging.

The microstructures consist of the elongated $\alpha_1$ particles formed during TMT as well as small ($\simeq$70Å diameter in (a) and $\simeq$175Å in (b)) $\alpha_1$ particles formed due to secondary decomposition during step aging as a result of high supersaturation of the $\alpha_2$ phase and the increased undercooling. Figs. 3(a) and (d) do not show the secondary decomposition. The $\alpha_1$ particles are smaller also. When the temperature is lowered in small steps, compositional differences occur between the two phases by diffusion, and due to the asymmetry of the phase diagram, one phase becomes richer in Cr and depleted in Fe and Co and vice versa.

The magnetic coercivity and remanences of the alloys corresponding to Fig. 3 are as follows: (a) $H_c \simeq 240$ Oe, $B_r \simeq 12.7$ kG, (b) $H_c \simeq 610$ Oe, $B_r \simeq 13.0$ kG, (c) $H_c \simeq 530$ Oe, $B_r \simeq 12.7$ kG, (d) $H_c \simeq 330$ Oe, $B_r \simeq 13.5$ kG.

The higher values of $B_r$ are due to particle alignment during TMT and $B_r$ remains almost unaffected by the subsequent step aging. However, the coercivity is affected by the step aging. The increased coercivity is due to the increased compositional differences produced during the step aging. Thus, a combination of TMT and step aging produces both a higher value of remanence and higher coercivity, and therefore a higher energy product $(BH)_{max}$.

Continuous cooling after TMT also produces similar results. Addition of V or V+Ti to the alloys does not affect the microstructure or the magnetic properties for similar aging treatments.

(iii) Magnetization Process: Fresnel and Focoult micrographs in Fig. 4 show the magnetic domain structure of the Alloy B after aging at 650°C for 50 hours. Both phases in the figure are magnetic. These micrographs suggest that the domain walls lie within the $\alpha_2$ matrix phase, and are curved around $\alpha_1$ phase. They lie along <100> directions, a consequence of the crystal anisotropy. The domain wall energy of $\alpha_2$ phase is lower than that of the $\alpha_1$ phase[7] and thus the walls lie in the $\alpha_2$ phase. The

micrographs suggest that the domain walls lying in the $\alpha_2$ phase are pinned by the more strongly magnetic $\alpha_1$ particles. The magnetization reversal process of the alloys is controlled by domain wall pinning as opposed to single domain hardening, as was demonstrated by Belli et al.[8]

B. Microstructure and Mechanical Properties

The mechanical test results for isothermally aged alloys are summarized in Figs. 5(a) and (b) for Alloys A and B, respectively. The results for Alloy C are intermediate to those of Alloys A and B. The yield stress or the stress at fracture (whichever occurs first) and the percent uniform elongation were measured using tensile specimens that were aged at 600°C and 640°C for various times up to 100 hours. The general features of the two curves show that the alloy aged at lower temperature has a greater increase in yield stress (over that of the unaged alloy), but this increase in strength is accompanied with a decrease in uniform elongation. In fact, Alloy A aged for 5 minutes at 600°C already failed before yielding took place, whereas Alloys B and C were measurably ductile for aging times up to 10 hours and 5 hours, respectively at 640°C. Aging at 640°C produced a modest increase in the yield strength with little change in the uniform elongation in all three alloys.

The variation in Vickers Micro-Hardness of Alloy A as a function of time at 600°C and 640°C is shown in Fig. 5(c). When aging is done at 640°C, there is a slight increase in the microhardness which rapidly saturates, similar to the tensile data. However, when aging is carried out at 600°C, the microhardness continues to increase over the first 10 hours and only slightly decreases at 100 hours in obvious contrast to the tensile data.

Examination of the fracture surfaces reveals a marked difference in the mode of failure between the materials aged at 600°C and at 640°C. When aged at 600°C, it has a mixture of dimpled rupture and grain boundary separation (see Fig. 6). Chemical analysis of the grain boundary

regions shows a definite Cr enrichment at the grain boundaries. Transmission electron microscopy and diffraction (Fig. 7) shows a second phase precipitating at the grain boundary which has been identified as the Cr-rich σ-phase.

The mechanical hardening of the aged alloys over unaged alloys is a consequence of the spinodal decomposition.[9] At lower aging temperature, the composition difference between the two decomposing phases is greater, leading to an increase in the hardness or yield stress as in Fig. 5. However, the material is severely embrittled after aging. The evidence of intergranular fracture from Fig. 6 and the presence of σ-phase in Fig. 7 suggest that the grain boundary embrittlement can be suppressed to some degree by a mechanical deformation so that the film of σ-phase breaks up into small particles. Also, the data suggest that replacement of 5% Cr with either V or V+Ti suppresses the embrittlement to some extent. This is partially due to smaller grain size resulting from lower homogenization temperature. Grain sizes for Alloys A and B are 500μ and 50μ, respectively. Also, Cr is known to cause enhanced embrittlement.[10] Thus, the Cr-rich $\alpha_2$ phase possibly controls the loss of ductility and addition of V or V+Ti can suppress fracture.

## IV. Conclusion

From the magnetic data and correlation with the microstructure, it can be concluded that for Fe-Cr-Co alloys, TMT combined with step aging or continuous cooling produces optimum magnetic properties. The microstructural features for high B are developed by TMT and the coercivity is developed by step aging and continuous cooling. The morphology of the microstructure is very much affected by the aging temperature. The alloy compositions do not affect the magnetic properties. However, from the mechanical property data, it is seen that mechanical properties deteriorate severely with aging. Alloying with V or V+Ti improves the mechanical properties and it is suggested that a plastic deformation step may help further.

Acknowledgement

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## FIGURE CAPTIONS

Fig. 1 B.F. micrographs of Alloy A isothermally aged for 1 hour at a) 670°C, b) 650°C, c) 640°C.

Fig. 2 B.F. micrographs of Alloy B a) after aging at 650°C for 1 hour, b) after TMT at 650°C for 1 hour in a field of 2kOe.

Fig. 3 B.F. micrographs of Alloy A step-aged after thermomagnetic treatment for 1 hour at A) 670°C, B) 660°C, C) 650°C, and D) 640°C.

Fig. 4 Fresnel (a) and Focoult (b) micrographs of Alloy B isothermally aged at 650°C for 50 hours, showing domain wall (c) lying in the $\alpha_2$ phase.

Fig. 5 Mechanical property vs. aging times for Alloys (a) A and (b) B aged at different temperatures. (c) Shows the Vicker's hardness for Alloy A.

Fig. 6 Fractographs from Alloy A aged at a) 640°C and b) 600°C. Note the intergranular fracture in (a) and both inter- and intra-granular fracture in (b).

Fig. 7 Bright field TEM micrograph from Alloy B showing σ-phase particles along the grain boundary.

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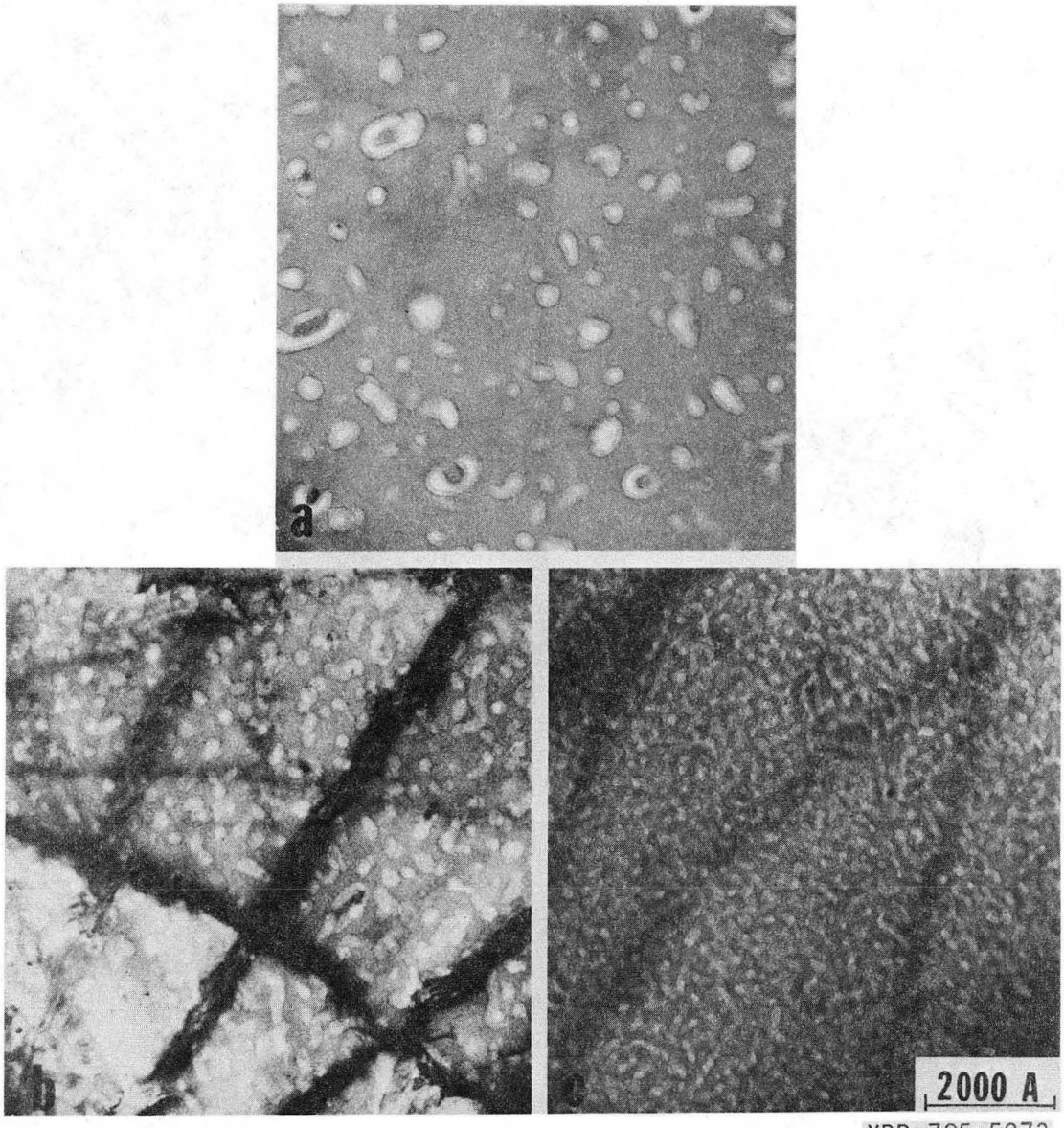


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Fig. 1

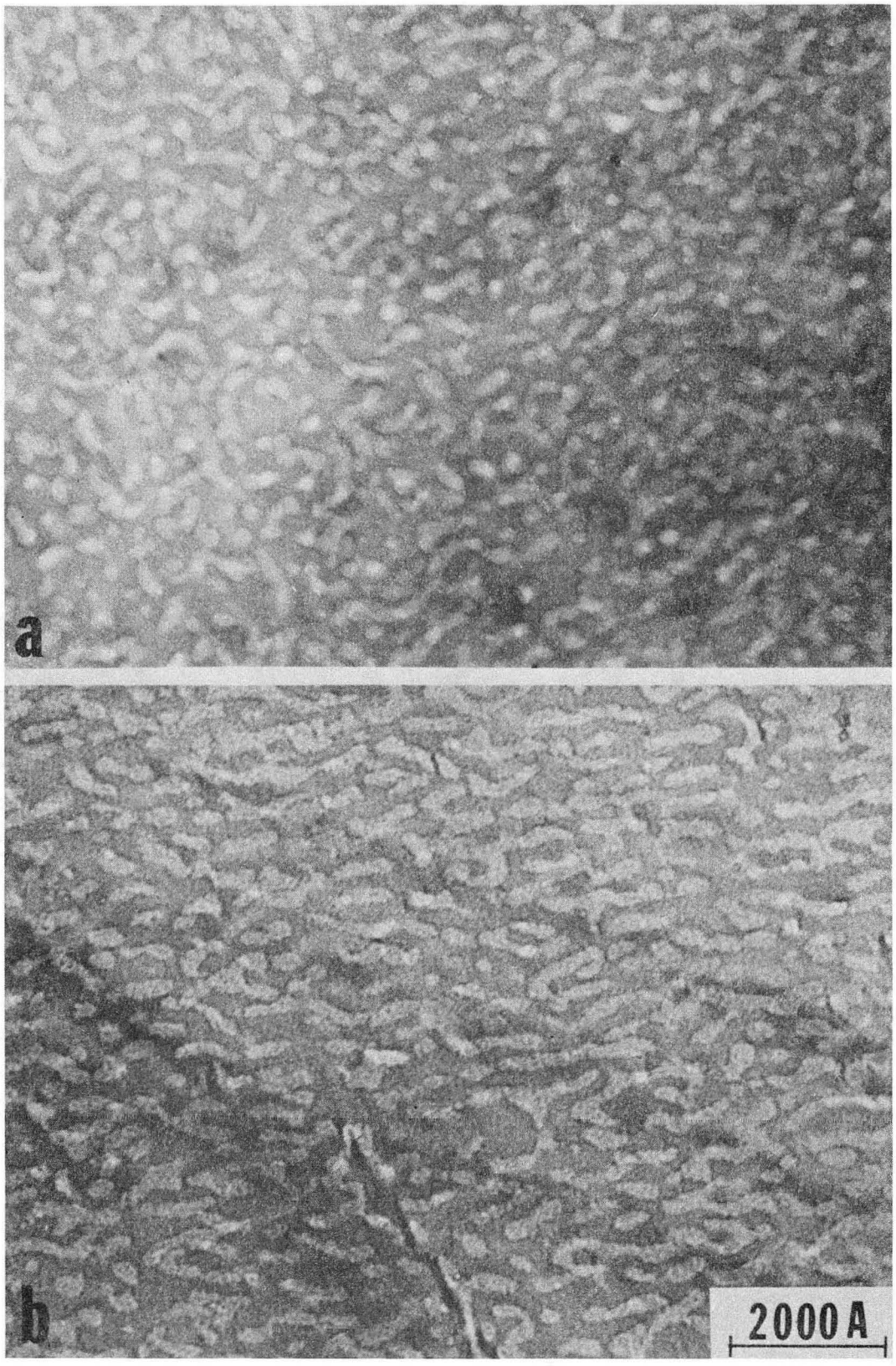


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Fig. 2

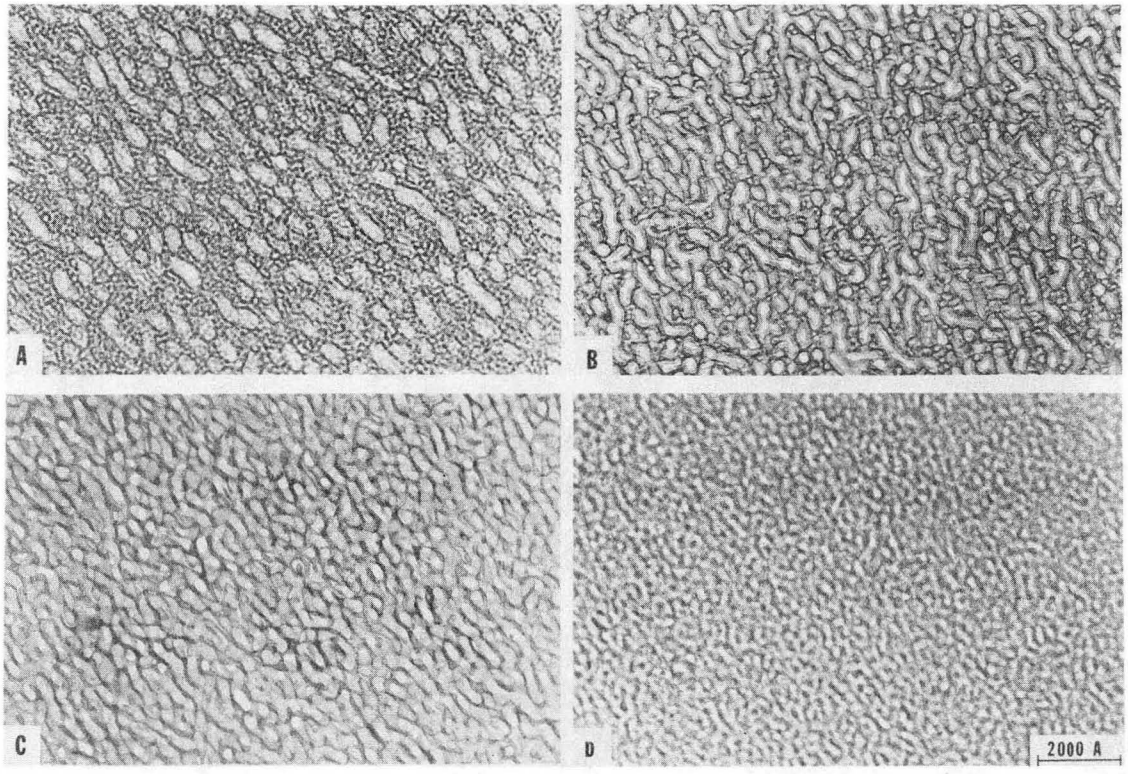


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Fig. 3

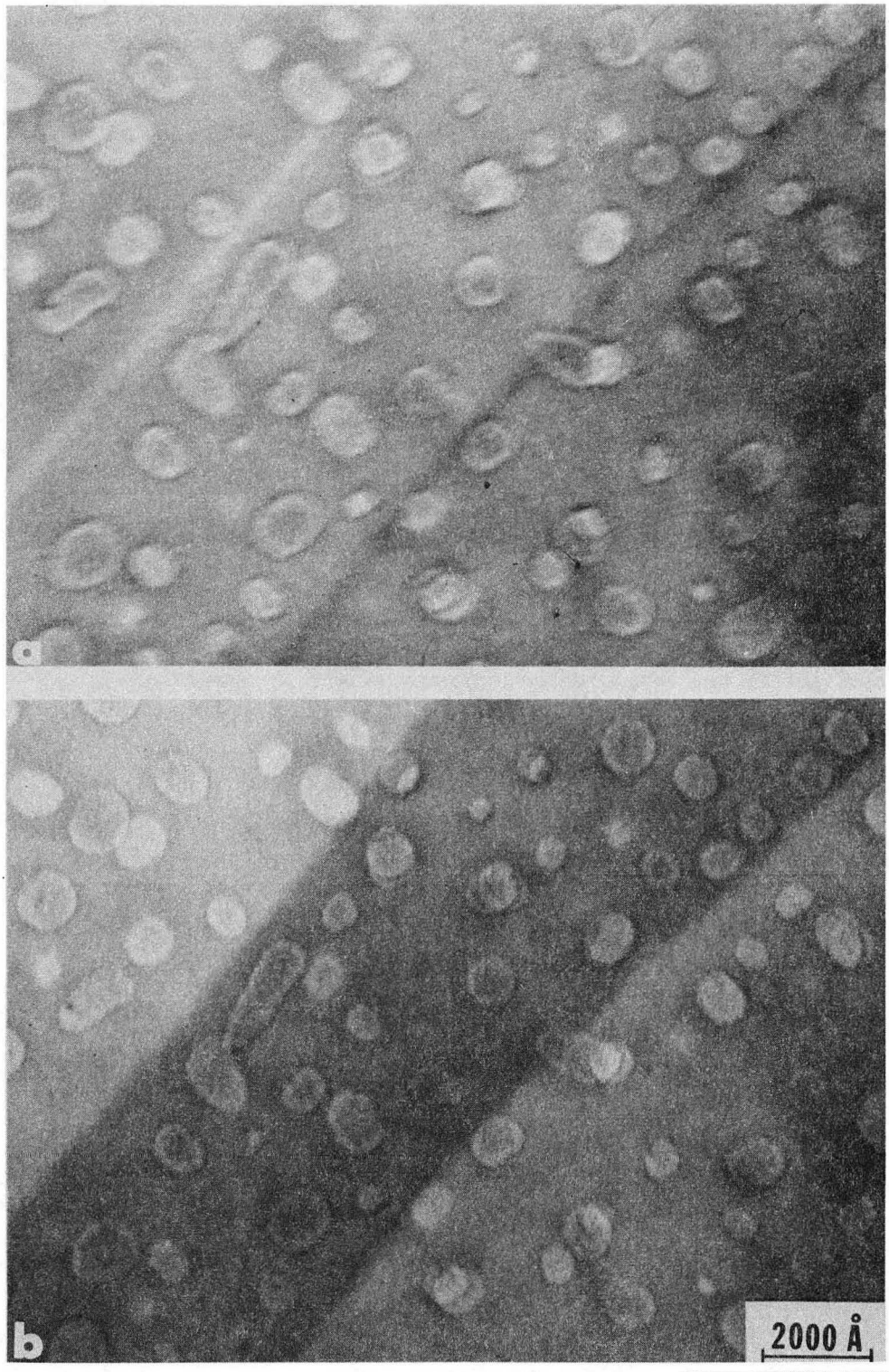


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Fig. 4

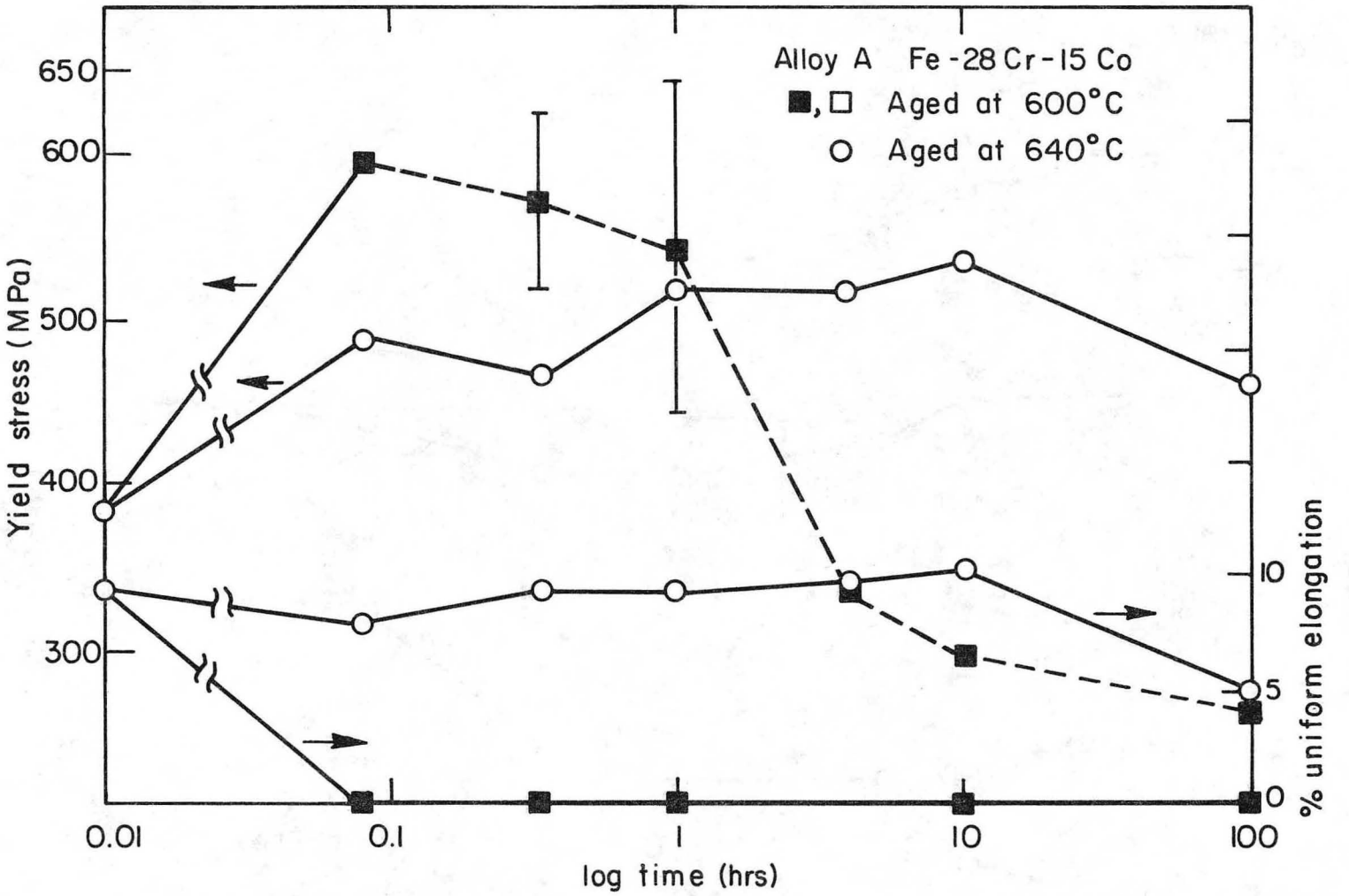
Alloy A Fe-28 Cr-15 Co
■, □ Aged at 600°C
○ Aged at 640°C
650
600
500
400
300
Yield stress (MPa)
10
5
0
% uniform elongation
0.01
0.1
1
10
100
log time (hrs)
XBL788-5569

Fig. 5(a)

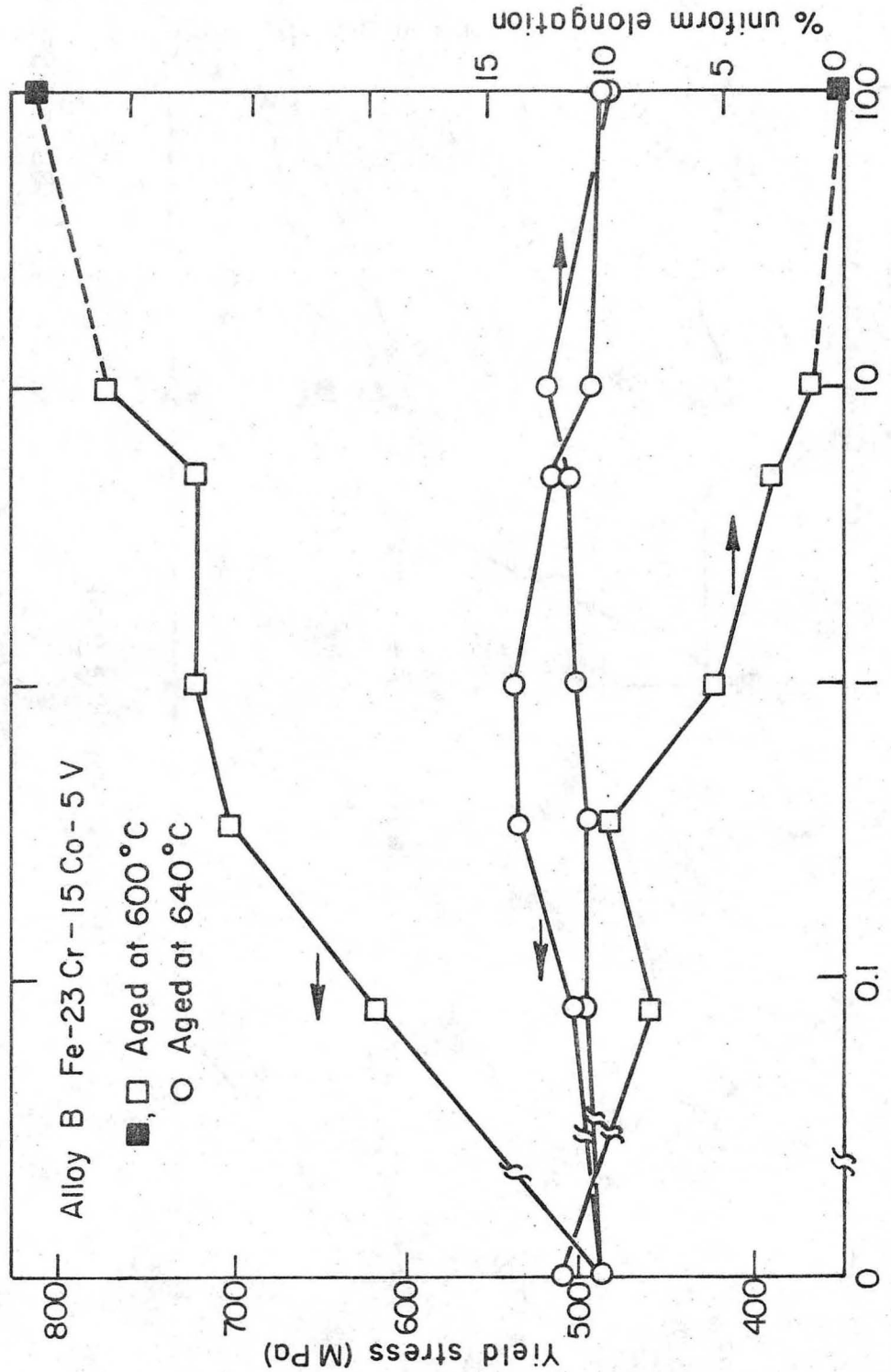


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Fig. 5(b)

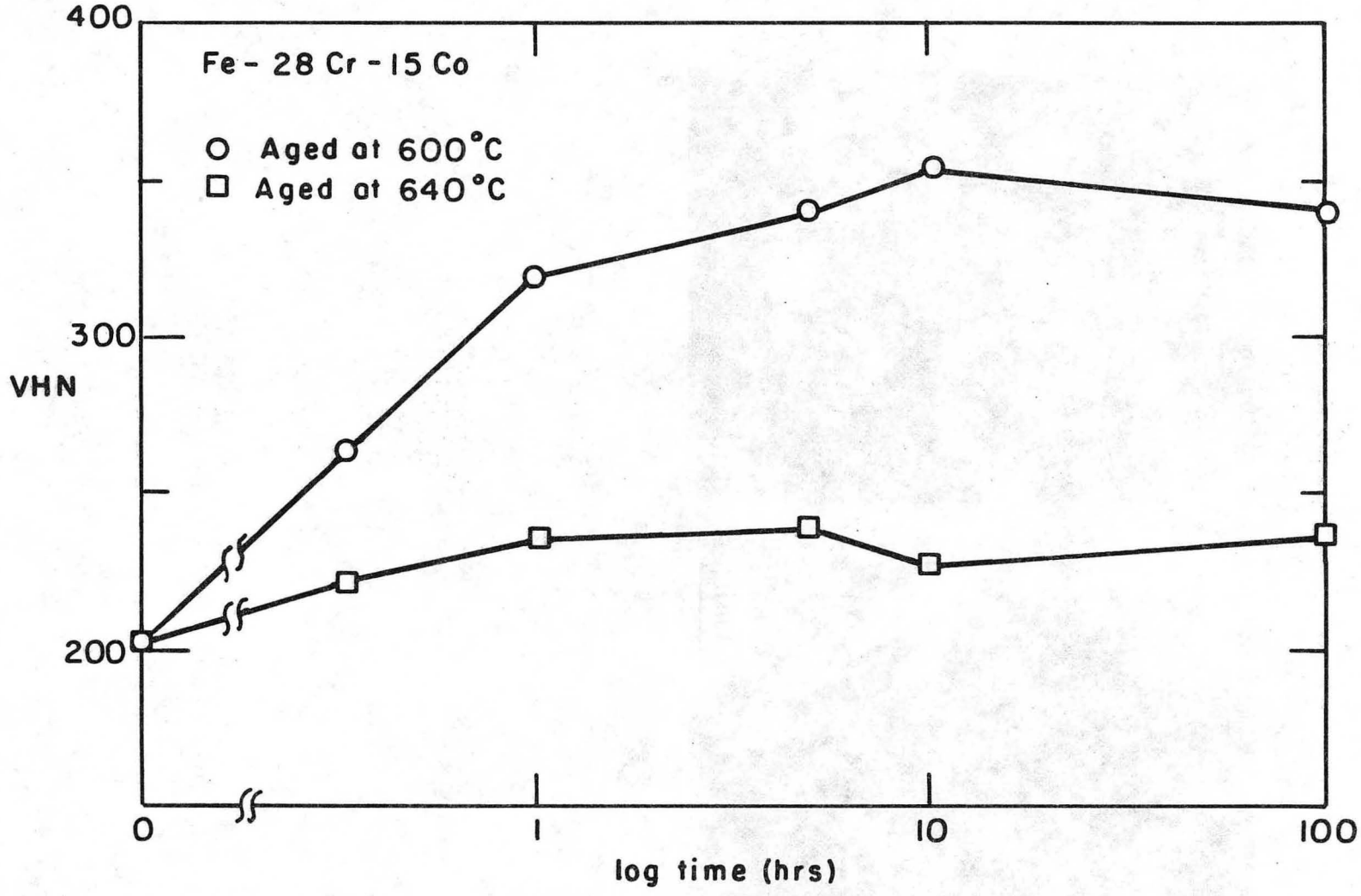


Fig. 5(c)

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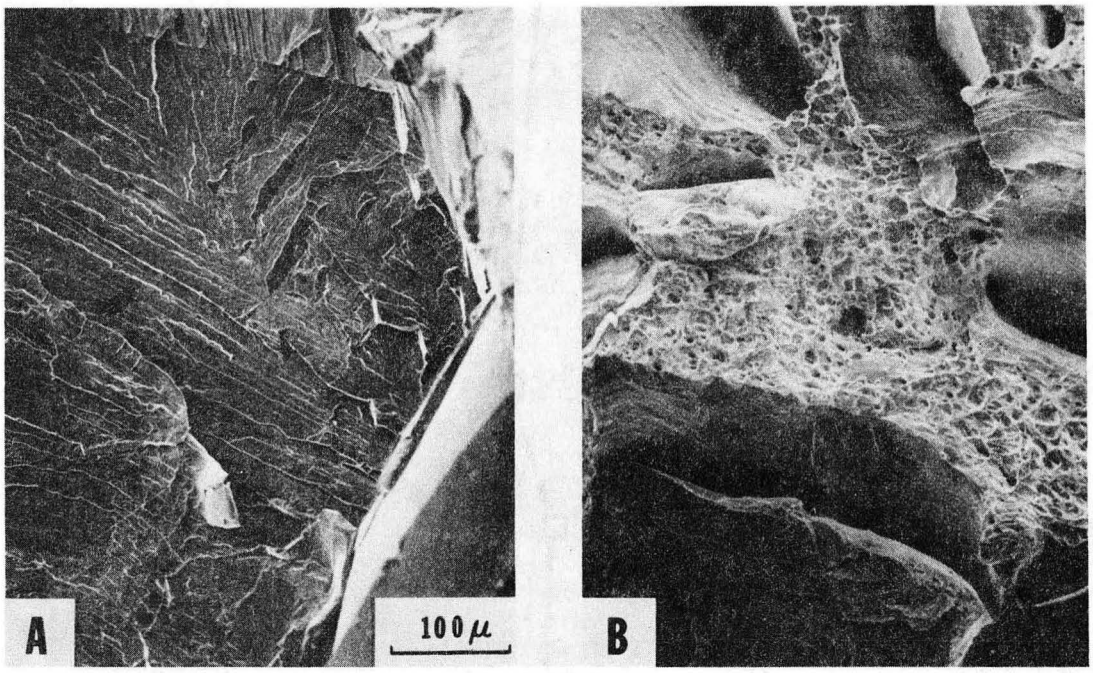


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Fig. 6

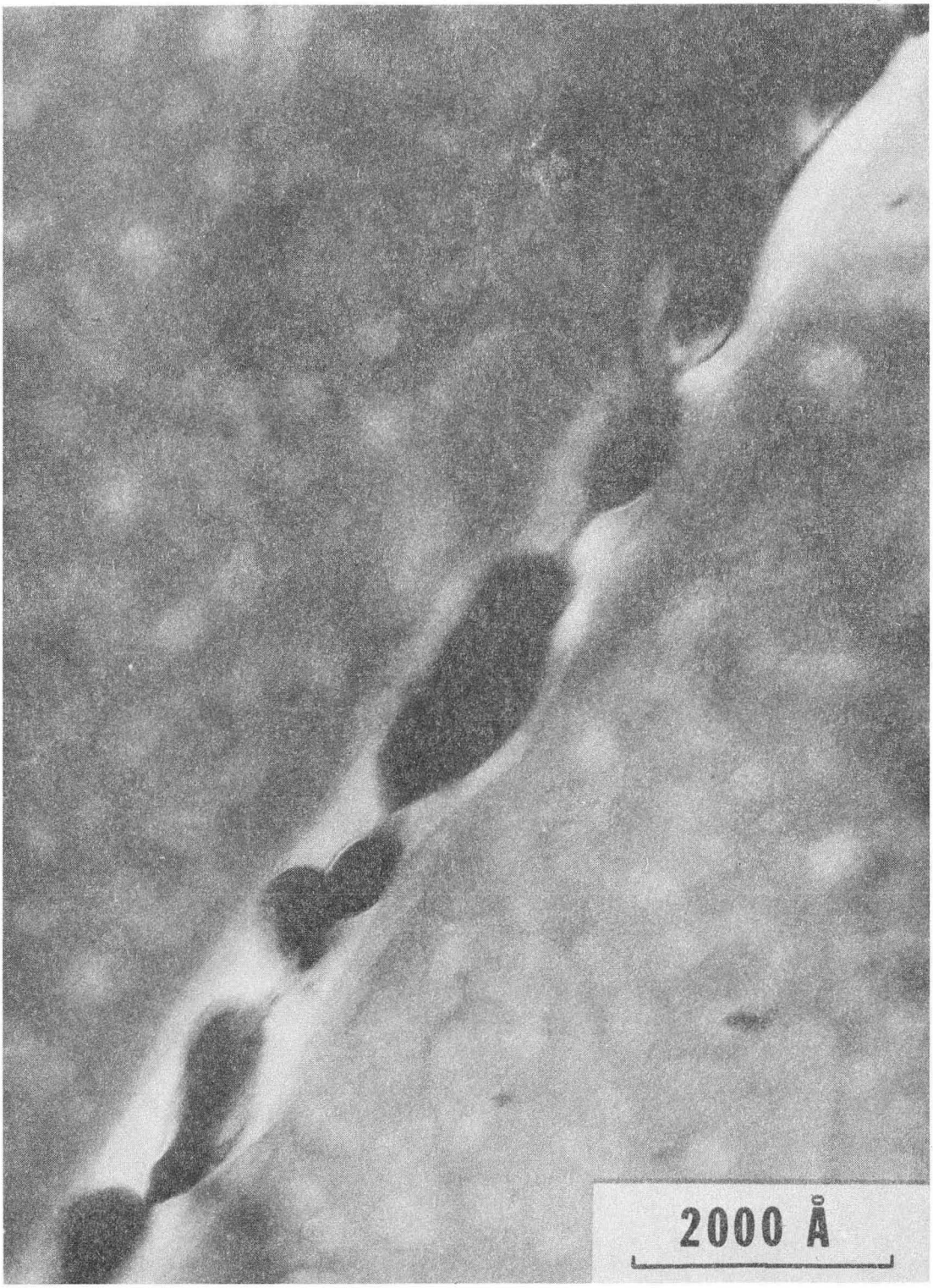


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Fig. 7

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