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AN IRON-NICKEL-TITANIUM ALLOY WITH OUTSTANDING TOUGHNESS AT CRYOGENIC TEMPERATURE

by

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ABSTRACT

Recent research has shown that the alloy Fe-12Ni-0.25Ti may be processed to have an outstanding combination of strength and toughness at cryogenic temperatures. In this paper we present some of the results obtained to date with this alloy, and compare these to the properties of two commonly used cryogenic steels. Tensile properties, impact properties, and preliminary results of fracture toughness tests are presented.

I. INTRODUCTION

Recent research in this laboratory has led to the identification of a group of ferritic alloys from the iron-nickel-titanium system which show an unusual combination of strength and ductility at cryogenic temperatures. The details of this research are reported elsewhere(1). In this paper we shall present some of the best results obtained through processing of an Fe-12Ni-0.25Ti alloy and compare these to the best available properties of two commonly used cryogenic steels: type 304 stainless steel and Fe-9Ni-1Mn-0.1C ("9-Nickel" steel).

II. ALLOY COMPOSITION AND PROCESSING

Low carbon alloys of nominal composition Fe-12Ni-0.25Ti were obtained by vacuum melting from the prime components. The actual composition of typical ingot is shown in Table I. The 2-3/4" ingots were forged to 3/4" plate at a temperature of approximately 1100°C and air cooled to room temperature. The alloys were then treated to refine grain size and impart a beneficial distribution of fine coherent precipitate particles. The treatments involve annealing within the two-phase region, α (BCC ferrite) + γ(FCC austenite), which falls roughly at temperatures of 600-800°C for this alloy, in sequence with mechanical working or precipitation anneal at lower temperature.

The four processing sequences which will be specifically mentioned below are: (1) 900°C (2 hrs.), air quench + 700°C (2 hrs.) air quench + 550°C (2 hrs.), water quench; (2) 700°C (2 hrs.), air quench + 30% cold work + 700°C (2 hrs.), air quench + 550°C (2 hrs.), water quench;

(3) 700°C (2 hrs.), air quench (repeated four times) + 550°C (2 hrs.), water quench; (4) 900°C (2 hrs.), air cool + [700°C (2 hrs.), air quench + 660°C (2 hrs.), air quench] (repeated four to six times) + 550°C (2 hrs.) water quench. Treatment (1) imparts outstanding impact toughness at liquid nitrogen·temperature. Treatment (2) yields an alloy having outstanding impact toughness to temperatures below 6°K. Treatment (3) also imparts very good impact toughness at 6°K, while avoiding the cold-working step. Treatment (4) yields an alloy of extremely fine grain size (< 1 micron) which shows exceptional ductility in fracture toughness tests at liquid nitrogen temperature.

In succeeding sections the mechanical properties of this alloy will be compared to those of type 304 stainless steel and "9-nickel" steel. The comparison alloys were procured commercially and treated according to recommended procedures(2) to optimize toughness at cryogenic temperature. The type 304 stainless steel was austenitized at 1020°C for one hour, then ice brine quenched. The "9-nickel" steel was processed in the sequence: 900°C (2 hrs.), air quench + 790°C (2 hrs.), air quench + 550°C (2 hrs.), water quench.

III Cryogenic Tensile Properties

The tensile properties of the Fe-12Ni-0.25Ti alloy, treatments 1, 2, and 3, were tested at 7°K, in an Instron machine equipped with a liquid helium cryostat using specimens of 0.5 in. guage length and 0.125 in. diameter. The tensile properties of the two comparison alloys were measured under identical conditions.

The results are given in Table II. The Fe-Ni-Ti alloy, treatments 2 and 3 showed virtually identical tensile properties. While the yield strength of these alloys is slightly below that of the 9Ni steel, they are distinguished by their high true stress at fracture (700 ksi at 7°K) and their excellent ductility (77% reduction in area at 7°K).

IV. CRYOGENIC IMPACT TOUGHNESS

Charpy impact tests were conducted at liquid nitrogen temperature using ASTM standard techniques and at 6°K using the "boxing" technique described elsewhere in these proceedings(3). The Fe-12Ni-0.25Ti alloy was tested in each of the four processing conditions described in Section II. The 304 and 9-Nickel comparison alloys were also tested.

The results of the Charpy impact tests are shown in Table III. At 77°K the Fe-12Ni-0.25Ti alloy showed outstanding impact toughness in each of the four treatments. The Charpy energy of the alloy processed in sequence (2) exceeded the capacity of our test machine. When the test temperature was lowered at 6°K the alloy processed through sequence (1) became relatively brittle, having a Charpy energy below those of the 304 and 9-Nickel steels. Alloys processed through sequences (2), (3) and (4), however, retained high impact toughness. The Charpy energy obtained through treatment (2) again exceeded the capacity of the machine.

The results of these tests are illustrated in Figures 1 and 2. In Figure 1 the impact fracture surface of the Fe-12Ni-0.25Ti alloy, processed through sequence (3), is compared to the fracture surfaces of the 304 and 9-Nickel steels. The higher impact toughness of the Fe-12Ni-0.25Ti alloy is apparent in the severe deformation of its fracture surface. Figure 2 contains a photograph of a sample of the

Fe-12Ni-0.25Ti alloy processed through sequence (2) and tested at 6°K. The impact fracture propagated through roughly 90% of the thickness of the bar before the hammer was stopped.

V. FRACTURE TOUGHNESS

While we have completed only preliminary fracture toughness testing to date, results confirm the unusual ductility of the processed Fe-12Ni-0.25Ti alloy. Fracture toughness tests were conducted at 77°K on an MTS machine equipped with a liquid nitrogen cryostat. The specimens were ASTM standard "compact tension" specimens(4) of 0.70 in. thickness. Alloys tested included Fe-12Ni-0.25Ti, treatments (1) and (4), and 9-Nickel steel.

The results of these test are shown in Figure 3, which includes the load-displacement curves obtained. As may be seen from the shape of the curves none of the alloys tested was actually in a plane strain loading condition at "pop-in" (unstable crack propragation as indicated by the sudden drop in the load-displacement curve). However, the 9-Nickel steel was in nearly plane strain loading (its load-displacement curve approximates to a straight line) and exhibits a fracture toughness value (K_Q) in the range 130-150 ksi $\sqrt{in.}$ The Fe-12Ni-0.25Ti alloy, processed through sequence (4) was virtually immune to unstable crack growth in this test. The induced crack in this sample grew in a stable manner with marked concommitant plastic deformation until the test was finally terminated.

The three fracture toughness specimens are compared in Figure 4. The enchanced ductility of the Fe-12Ni-0.25Ti alloys is visually apparent. Scanning electron fractographs of the fracture surfaces of the three samples are presented in Figure 5. These fractographs were taken along the center-line of the samples slightly ahead of the front of the pre-induced fatigue crack. They show that crack propagation through the center of the 9-Nickel and the Fe-12Ni-0.25Ti, process (1) alloys occurred in a semi-brittle manner through quasi-cleavage. Crack propagation in the Fe-12Ni-0.25Ti, process (4) alloy was almost purely ductile. We found virtually no evidence of quasi-cleavage on the fracture surface.

Since the Fe-12Ni-0.25Ti alloys were well away from plane strain conditions during these test no fracture toughness measure was taken. Tests using thicker samples are currently in progress.

VI. DISCUSSION

The results presented in the previous sections indicate that the Fe-12Ni-0.25Ti alloy can be processed to have an excellent combination of strength and toughness at very low temperatures. The impact toughness values in particular are well above any which, to our knowledge, have ever been obtained at cryogenic temperatures.

It is not yet clear whether the properties reported here are percuiliar to Fe-Ni-Ti alloys of high nickel content or whether they may be reproduced in less highly alloyed systems through proper processing. For example, reports of recent Japanese work on the 9-Nickel alloy(5) indicates that Charpy impact values as high as ~160 ft.-lbs. can be obtained at liquid nitrogen temperature through careful alloy preparation. Nor is it now clear whether the rather complex processing sequences we have used to obtain these properties can be simplified to processes of more commercial appeal. These questions are now being investigated.

ACKNOWLEDGEMENTS

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TABLE I

Chemical Analysis of Two Ingots of Nominal Composition Fe-12Ni-0.25Ti

	Ni	Ti	C	N	S	P	Mn	O	Fe
1.	12.14	0.24	0.01	0.003	<0.005	<0.005	<0.005		balance
2.	10.90	0.23	0.008	0.004	<0.005	<0.005	<0.005	0.020	balance

TABLE II

Results of Tensile Tests at 7°K

Alloy	Yield Strength (KSI)	Engineering Tensile Strength (KSI)	True Stress At Fracture (KSI)	Elongation (%)	Reduction of Area (%)
304	105	270	∿580	42	53
9-Nickel	208	231	∿475	21	59
Fe-12Ni-0.25Ti Treatment					
1	183	208	∿520	20	71
2,3	185	204	∿700	23	77

TABLE III

Results of Charpy V-Notch Tests at Cryogenic Temperature

Alloy	Energy Absorbed (ft-lbs)	
	77°K	6°K
304	128	130
9 - Nickel	92	75
Fe-12Ni-0.25Ti Treatment: 1	204	26
2	>225*	>225*
3	205	205
4	180	180

FIGURE CAPTIONS

Figure 1. Charpy specimens tested at 6°K using the method given in reference (3). The Fe-12Ni-0.25Ti alloy was processed according to treatment (3) described in Section II. The 304 and 9-Nickel steels were also processed as described in Section II.

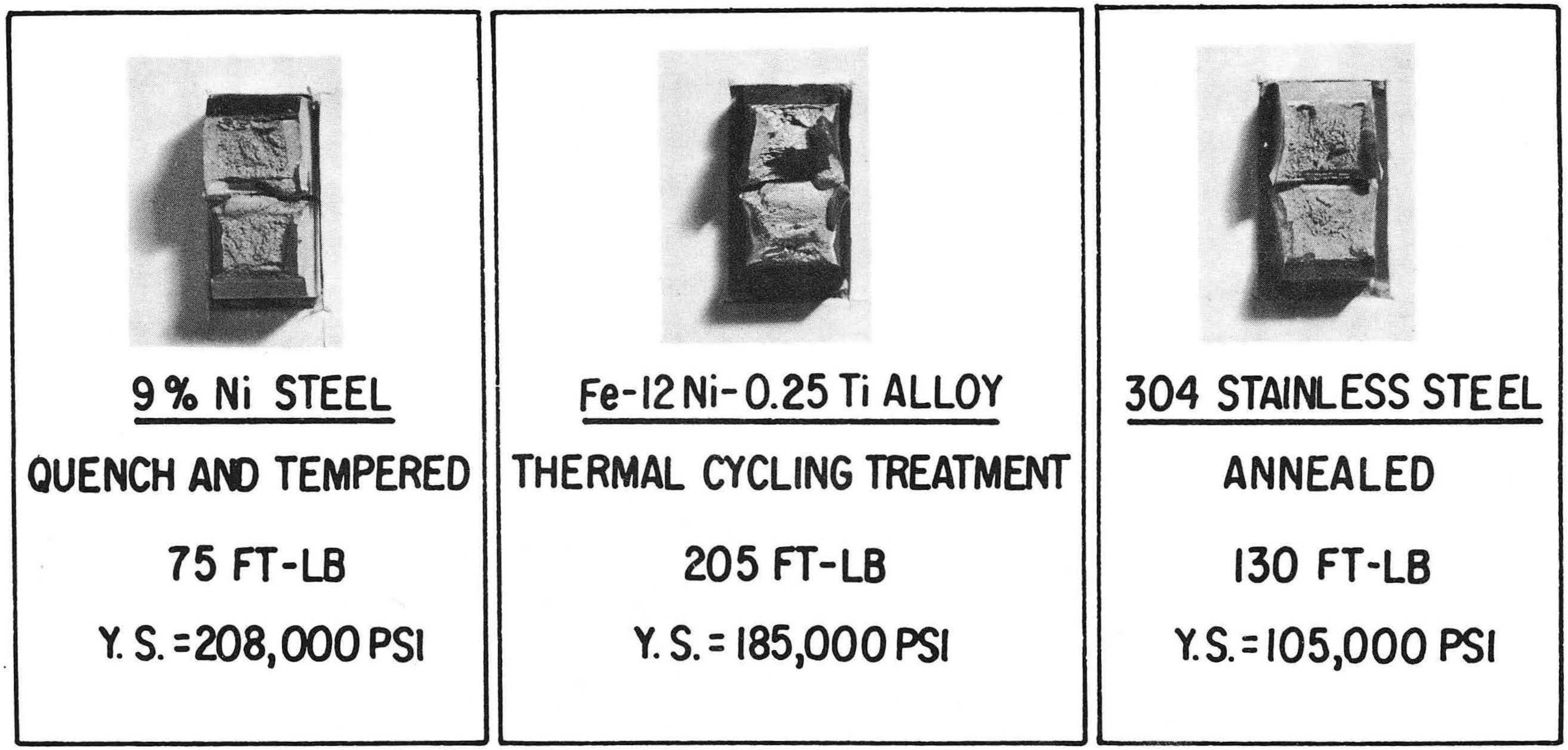
Figure 2. An Fe-12Ni-0.25Ti Charpy bar processed according to treatment (2) described in Section II and tested at 6°K using the procedure given in reference (3). The impact energy exceeds the capacity of the test machine (225 ft. lbs.).

Figure 3. Load-displacement curves obtained in fracture toughness tests comparing the 9-Nickel steel and two treatements of the Fe-12Ni-0.25Ti alloy at 77°K. The specimens were ASTM Standard "compact tension" specimens(4) 0.7 inch in thickness.

Figure 4. Post-test photographs of fracture toughness specimens: (a) 9-Nickel steel, (b) Fe-12Ni-0.25Ti, treatment (1), (c) Fe-12Ni-0.25Ti, treatment (4). The fracture in sample (c) propagated very slowly and the test was halted before the final fracture. The specimen has been cut open to reveal the fracture surface.

Figure 5. Scanning electron fractographs of the samples shown in fig. 4: (a) 9-Nickel steel, (b) Fe-12Ni-0.25Ti; treatment (1), (c) Fe-12Ni-0.25Ti, treatment (4). Fractographs (a) and (b) show the faceting characteristic of quasi-cleavage.

Fractograph (c) shows the "dimples" characteristic of ductile fracture. All micrographs are 400X magnification.



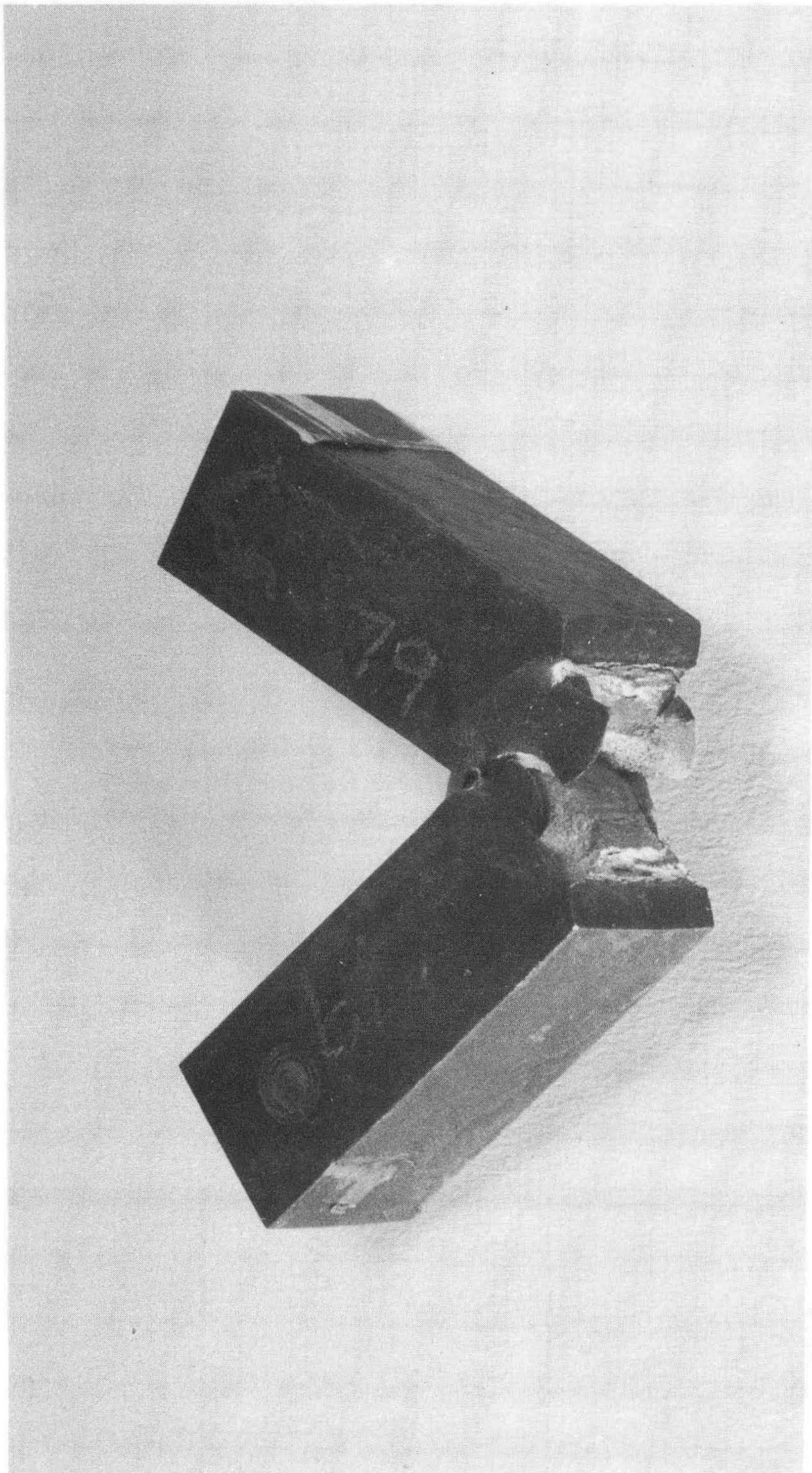
(a)

(b)

(c)

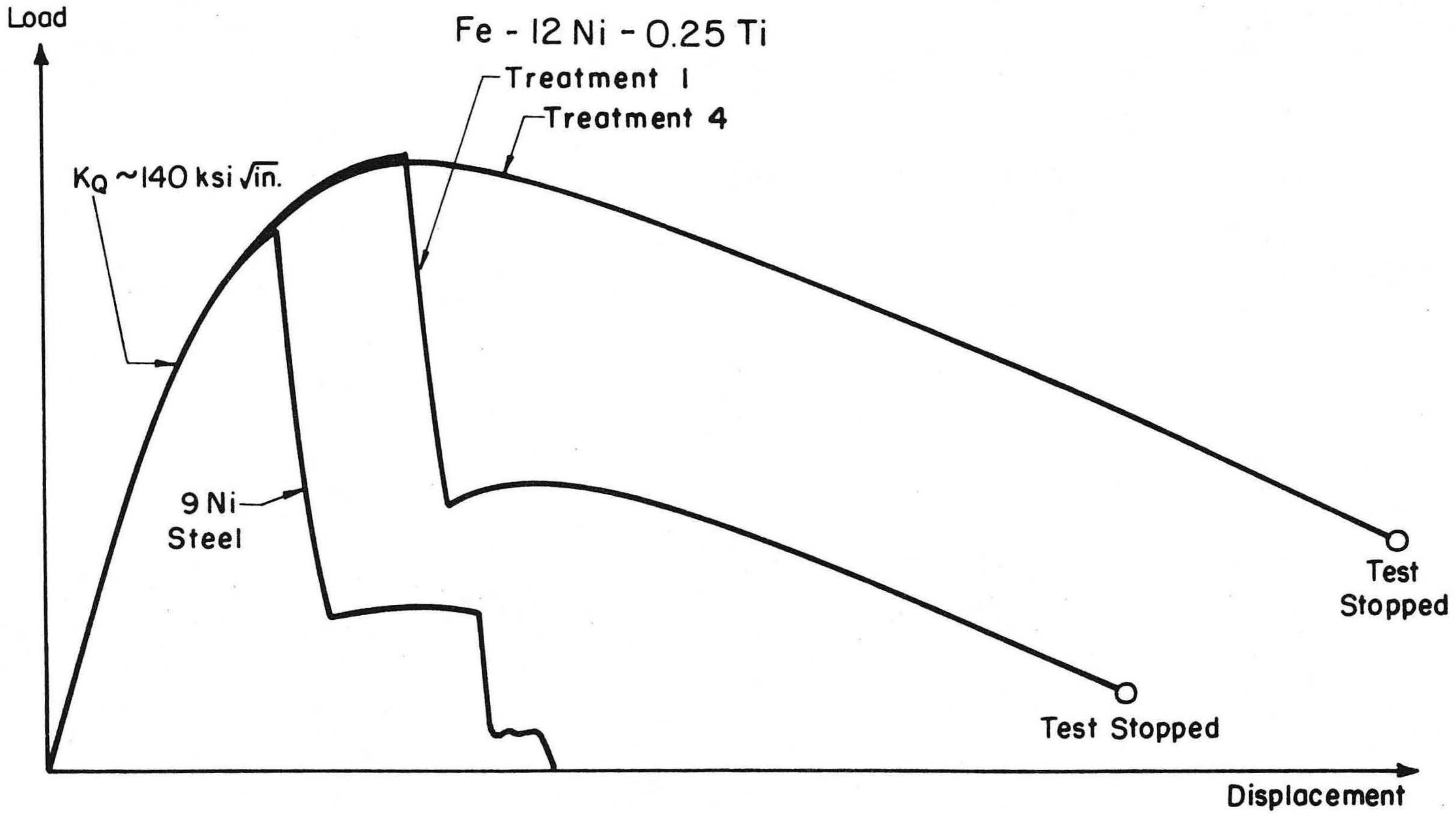
XBB 732-1216

Fig. 1



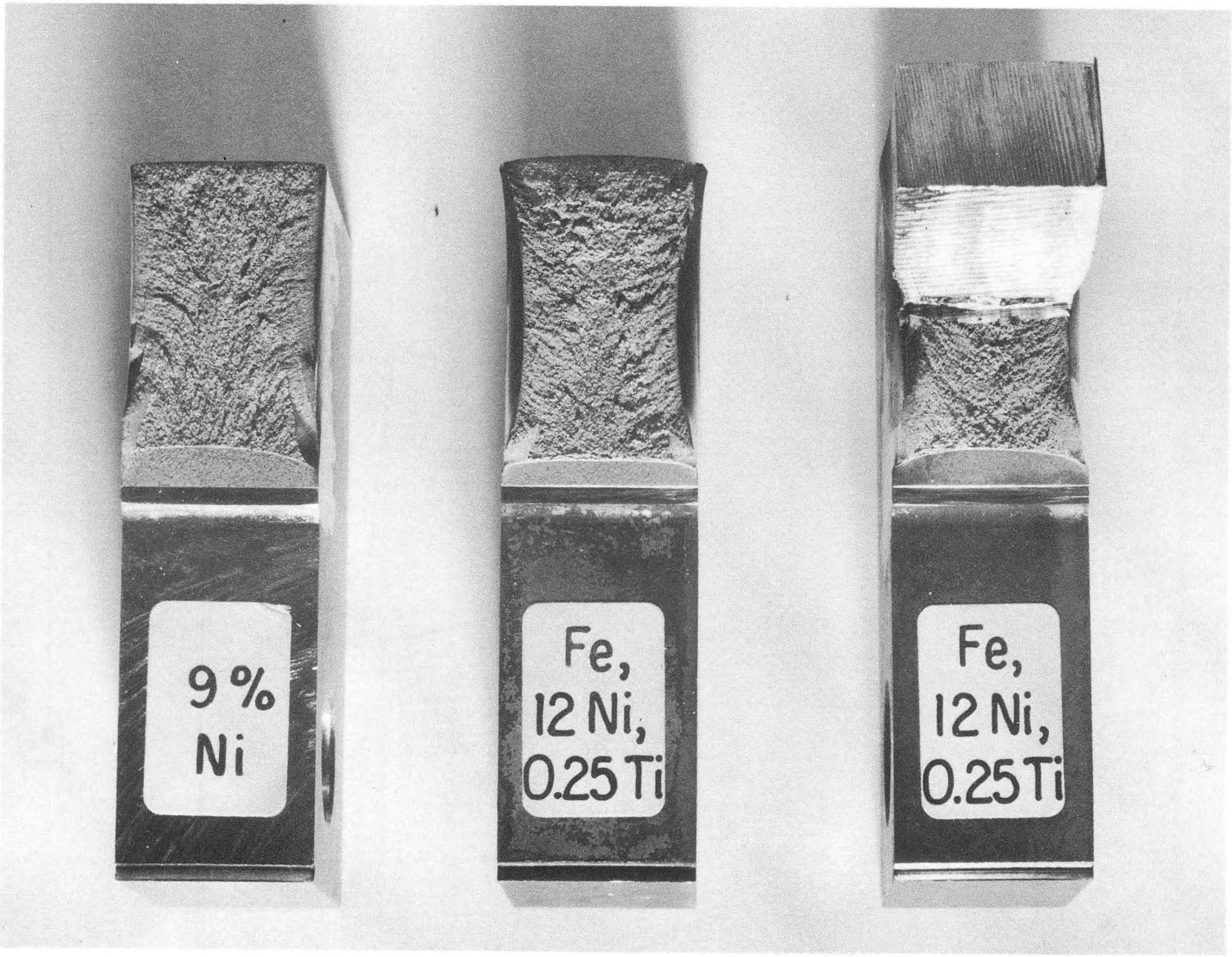
XBB 732-1215

Fig. 2



XBL 733-5855

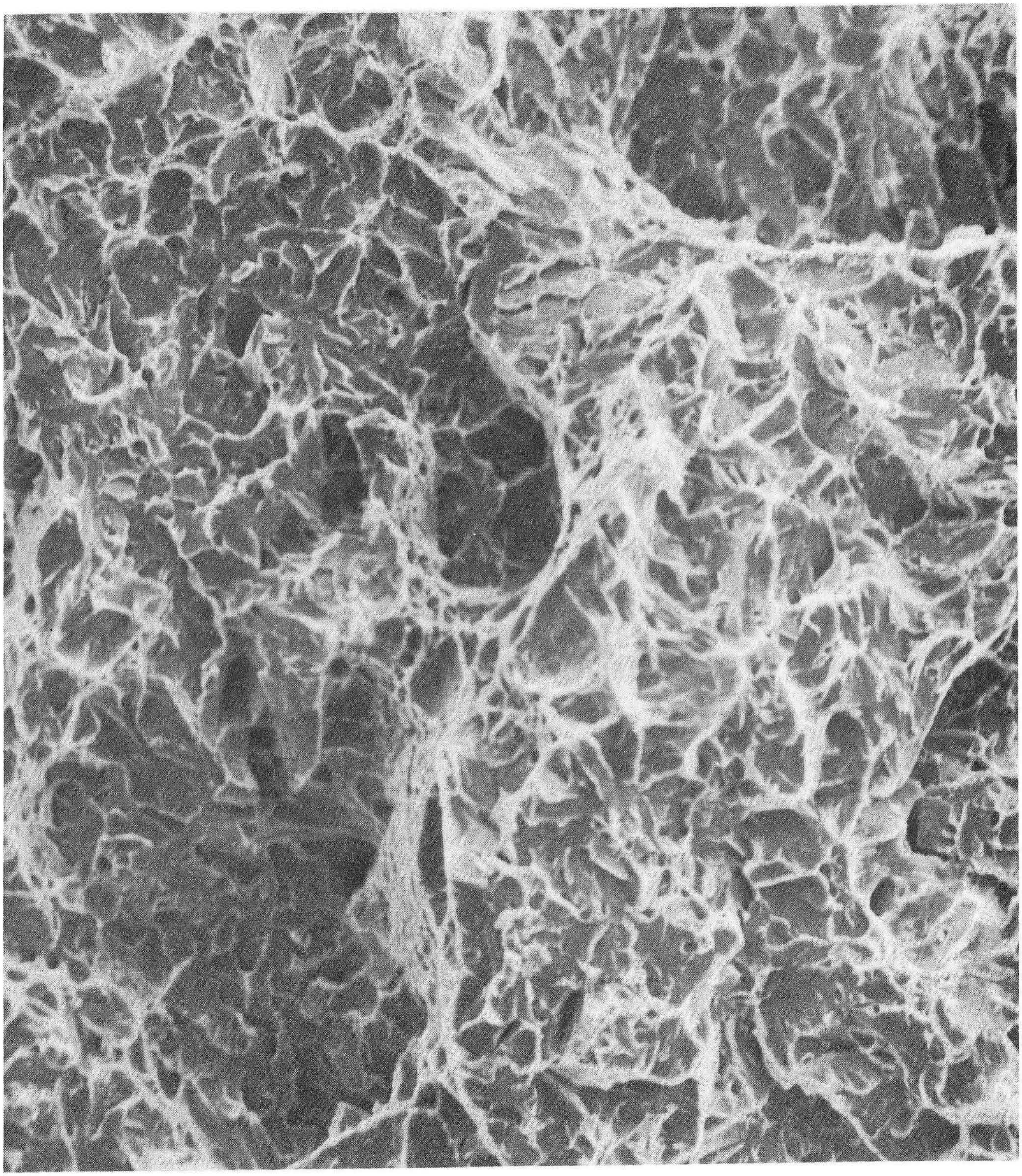
Fig. 3



(a) (b) (c)

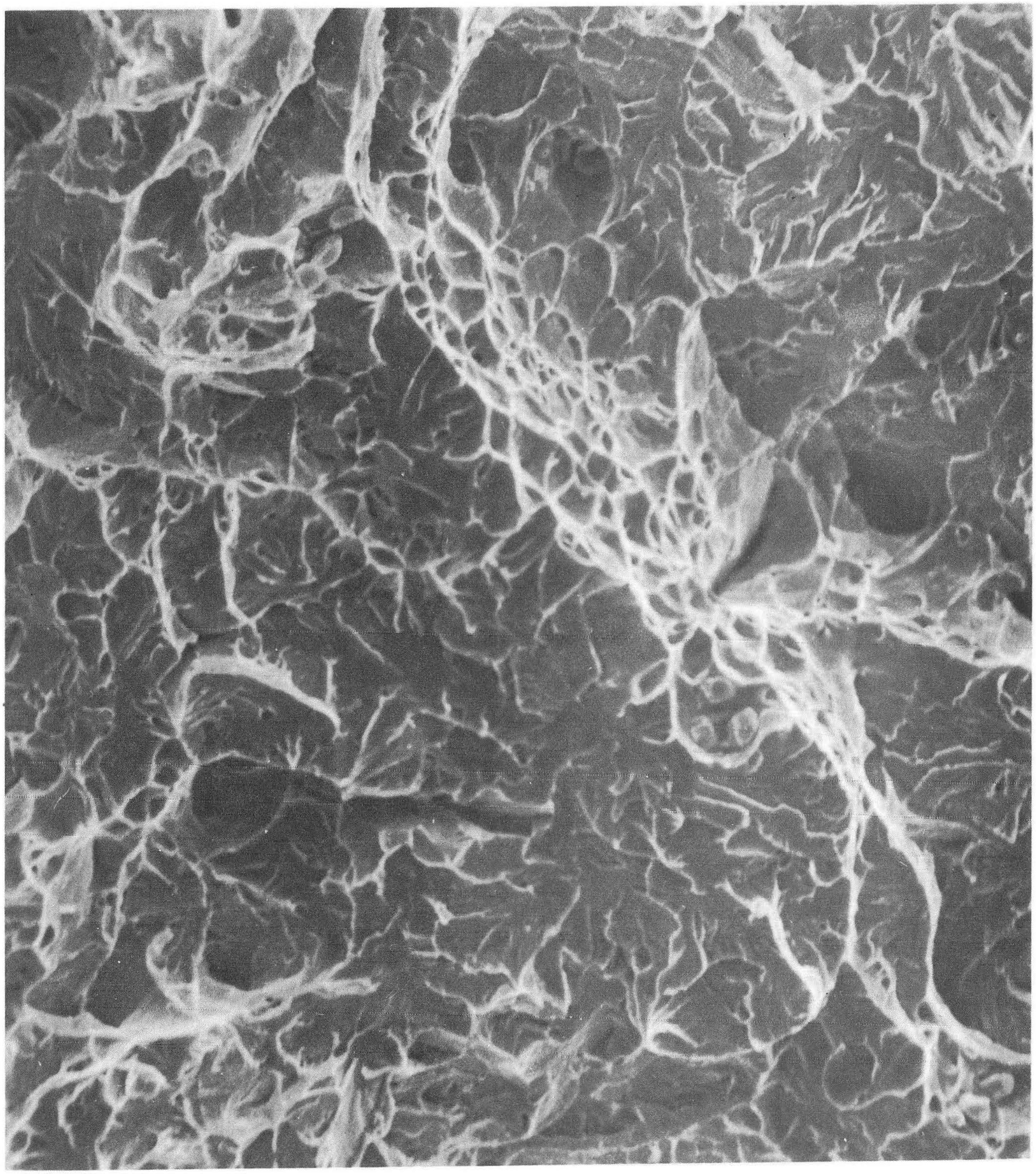
XBB 732-1220

Fig. 4



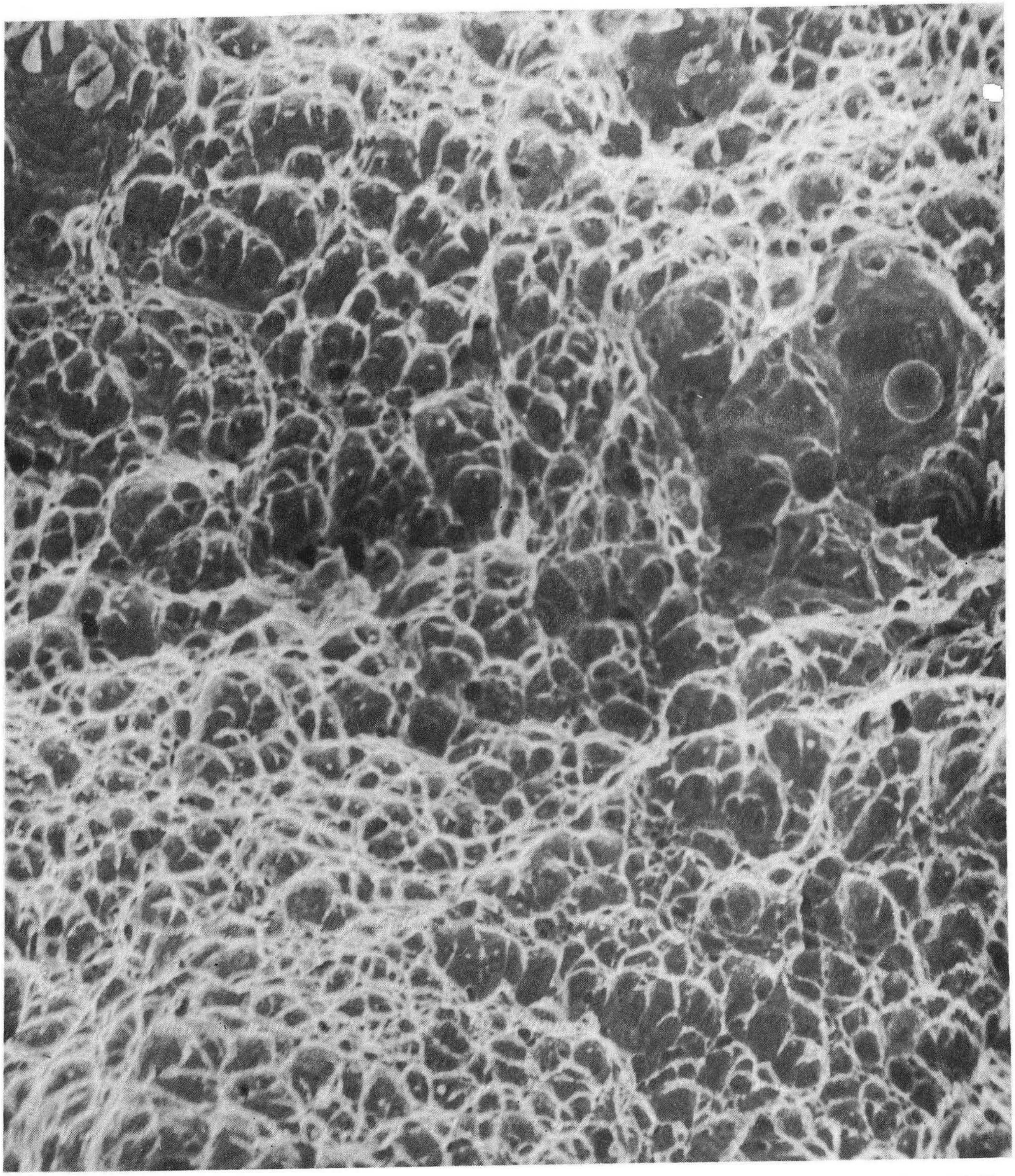
XBB 733-2165

Fig. 5a



XBB 733-2166

Fig. 5b



XBB 733-2167

Fig. 5c

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