To be presented at Cryogenic
Engineering Conf., Atlanta,
Georgia, Aug. 8-10, 1973

LBL-1484 Rev.

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S. Jin, J. W. Morris, Jr. and V. F. Zackay

July 1973

Prepared for the U.S. Atomic Energy Commission
under Contract W-7405-ENG-48

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

S. Jin, J. W. Morris, Jr. and V. F. Zackay

Department of Materials Science and Engineering, University of California, Berkeley and Inorganic Materials Research Division, Lawrence Berkeley Laboratory

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.

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).

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 compositions of two ingots are shown in Table I. Two 2-3/4 in. diameter ingots were homogenized at 1050°C for 80 hours and forged to 3/4 in. thick plates at a temperature of approximately 1100°C and air cooled to room temperature. The alloys were then heat treated in γ-phase region (above ~700°C) and the two phase ($\alpha+\gamma$) region (below ~700°C) in sequence with cold working or isothermal decomposition at 550°C.

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.

CRYOGENIC TENSILE PROPERTIES

The tensile properties of the Fe-12Ni-0.25Ti alloys (treatments 1, 2, 3 and 4) were tested at both 77°K and 6°K. The tests were conducted in an Instron machine equipped with either a liquid nitrogen cryostat (77°K tests) or a liquid helium cryostat (6°K tests). Specimens of 0.5 in. gauge length and 0.125 in. gauge diameter were tested using the Instron crosshead speed of 0.02 in./minute. The tensile properties of the two comparision alloys, 9-Ni steel and 304 stainless steel, were measured under identical conditions.

The results of the tensile tests at 77°K are given in Table II. The test results at 6°K are shown in Table III. At liquid nitrogen temperature the Fe-12Ni-0.25Ti alloys, treatments 1, 2, 3 and 4, showed virtually identical tensile properties. The yield strength of this alloy is about the same as that of the 9-Ni steel but the ductility is better as can be seen in Table II. At liquid helium temperature, however, the yield strength of Fe-12Ni-0.25Ti alloys is slightly below that of 9-Ni steel. This is probably due to the smaller amount of carbon in Fe-12Ni-0.25Ti alloys (Table I) than in 9-Ni steel which contain approximately 0.1% carbon. The Fe-12Ni-0.25Ti alloys are distinguished by their excellent ductility (between 66~77% in reduction area at 6°K).

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 above. The 304 and 9-Nickel comparison alloys were also tested.

The results of the Charpy impact tests are shown in Table IV. 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 Figs. 1 and 2. In Fig. 1 the impact fracture surface of the Fe-12Ni-0.25Ti alloys, 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.

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) (specimens were prepared from Ingot II), and 9-Nickel steel.

The results of these tests are shown in Fig. 3, which includes the load-displacement curves obtained. As may be seen from the shape of the curves, none of the alloys tested were actually in a plane strain loading condition at "pop-in" (unstable crack propagation 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 Fig. 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 Fig. 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. Since crack propagation in the Fe-12Ni-0.25Ti alloys were well away from plane strain conditions during these tests, no fracture toughness measure was taken. Tests using thicker samples are currently in progress.

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 perculiar to Fe-Ni-Te alloys of high nickel low impurity content with

titanium's scavenging effect on interstitials 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

The work reported here was supported by the Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory and by the Office of Naval Research under Contract N00014-69-A-0200-1062, Nr 031-762.

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4. Annual Book of ASTM Standards, Vol. 31, E399-70T, ASTM, New York, 1970.
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0 0 0 0 3 9 0 2 7 4 1

Table I. Chemical analysis of two ingots of nominal composition Fe-12Ni-0.25Ti.

	Ni	Ti	C	N	S	P	Mn	O	Fe
I.	12.14	0.24	0.01	0.003	<0.005	<0.005	<0.005	0.014	balance
II.	10.90	0.23	0.008	0.004	<0.005	<0.005	<0.005	0.021	balance

Table II. Results of tensile tests at 77°K

Alloy	Y.S.* (ksi)	T.S. (ksi)	Elong. (%)	R.A. (%)	Ingot Used
304	81	235	47.3	65.6	
9-Ni Steel	146	172	29.6	66.8	
Fe-12Ni-0.25Ti Treatment					
1	142	153	31.0	71.2	I
2	141	155	32.3	75.8	I
3	138	151	33.8	78.5	I
4	141	163	33.1	76.2	II

*0.2% offset yield strength.

Table III. Results of tensile tests at 6°K.

Alloy	Y.S.* (ksi)	T.S.** (ksi)	Elong. (%)	R.A. (%)	Ingot Used
304	105	270	41.6	52.7	
9-Ni	208	231	21.2	59.1	
Fe-12Ni-0.25Ti Treatment					
1	184	197	21.7	68.0	I
2	183	199	22.3	70.2	I
3	180	196	24.1	73.6	I
4	181	205	22.8	70.4	II

* A serrated yielding behavior was observed in all the specimens tested at 6°K. This is a common phenomena in the tensile tests of iron-base alloys near the liquid helium temperature. The yield strength was taken from the first discontinuous yielding point.

** The tensile strength was obtained from the upper locus of discontinuous peaks in the stress-strain curve.

Table IV. Charpy V-notch impact tests at cryogenic temperatures

Alloy	Energy Absorbed (ft/lbs) 77°K	6°K	Ingot Used
304	128	130	
9-Ni Steel	92	75	
Fe-12Ni-0.25Ti Treatment			
1	204	26	I
2	>225*	>225*	I
3	205	203	I
4	140	134	II

*The capacity of the impact test machine was exceeded and the pendulum was stopped by the specimens.

FIGURE CAPTIONS

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

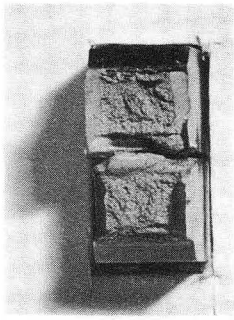
Fig. 2. An Fe-12Ni-0.25Ti Charpy bar processed according to treatment (2) described in the section of ALLOY COMPOSITION AND PROCESSING and tested at 6°K. The impact energy exceeds the capacity of the test machine (225 ft/lbs).

Fig. 3. Load-displacement curves obtained in fracture toughness tests comparing the 9-Nickel steel and two treatments of the Fe-12Ni-0.25Ti alloy at 77°K.

Fig. 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.

Fig. 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 400× magnification.

0 0 0 0 5 9 0 2 7 4 8



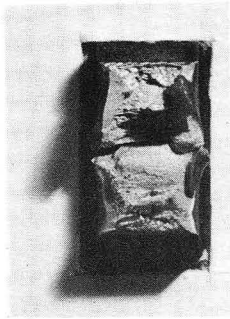
9 % Ni STEEL

QUENCH AND TEMPERED

75 FT-LB

Y. S. = 208,000 PSI

(a)



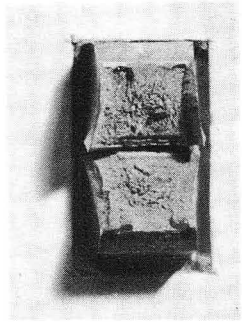
Fe-12 Ni-0.25 Ti ALLOY

THERMAL CYCLING TREATMENT

205 FT-LB

Y. S. = 185,000 PSI

(b)



304 STAINLESS STEEL

ANNEALED

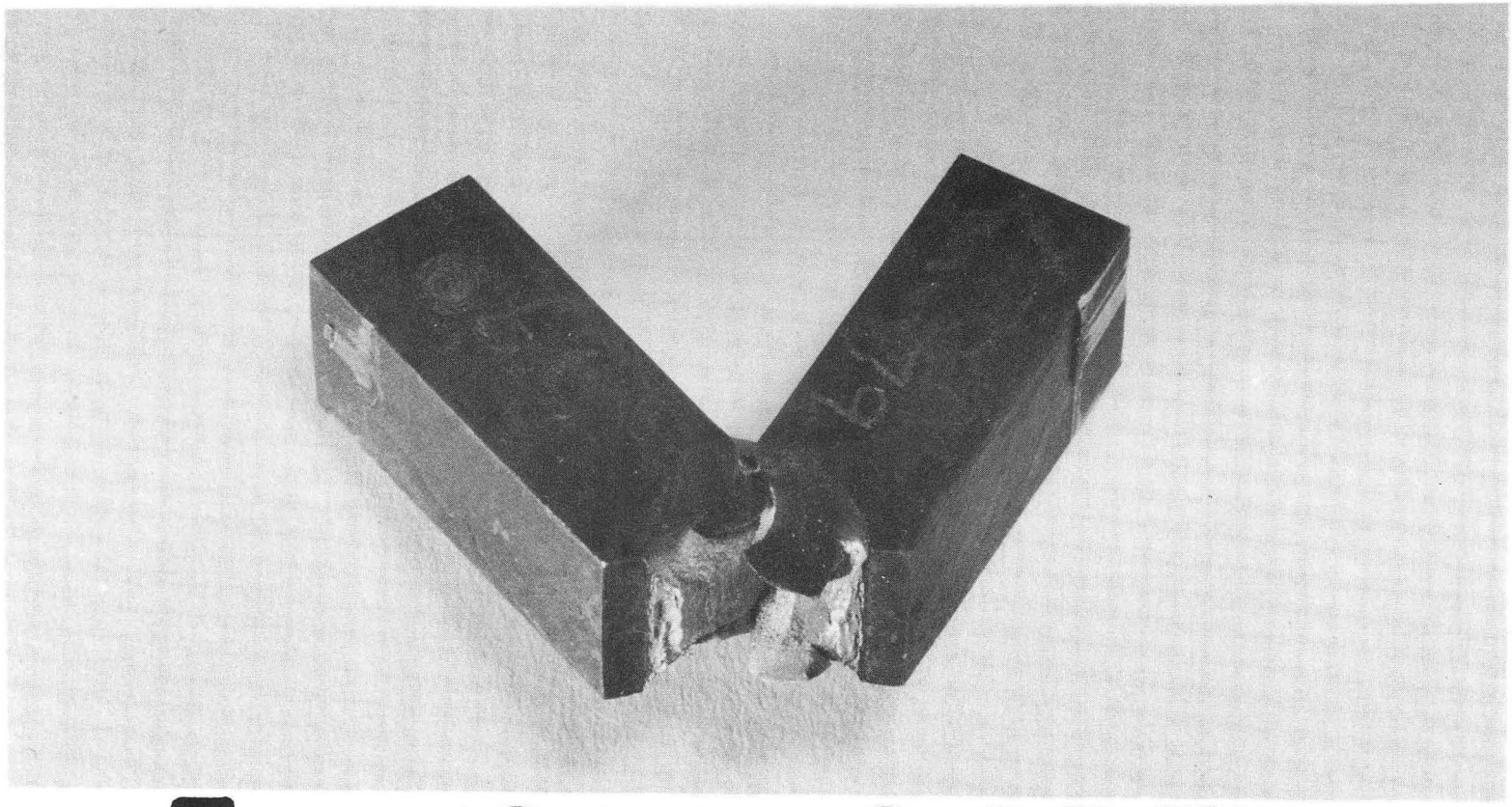
130 FT-LB

Y.S. = 105,000 PSI

(c)

XBB 732-1216

Fig. 1

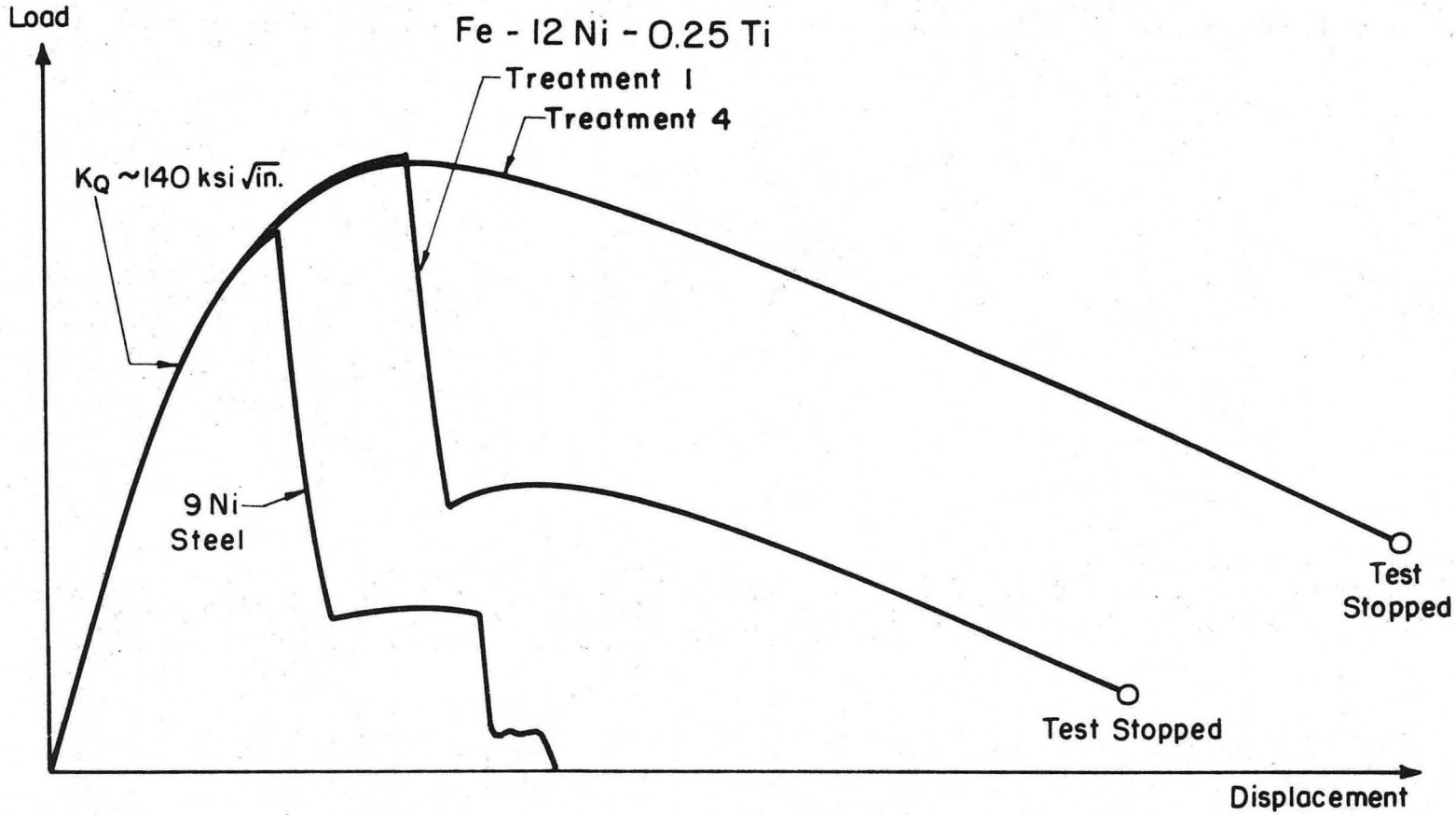


Fe - 12 Ni - 0.25 Ti

30% COLD WORKED

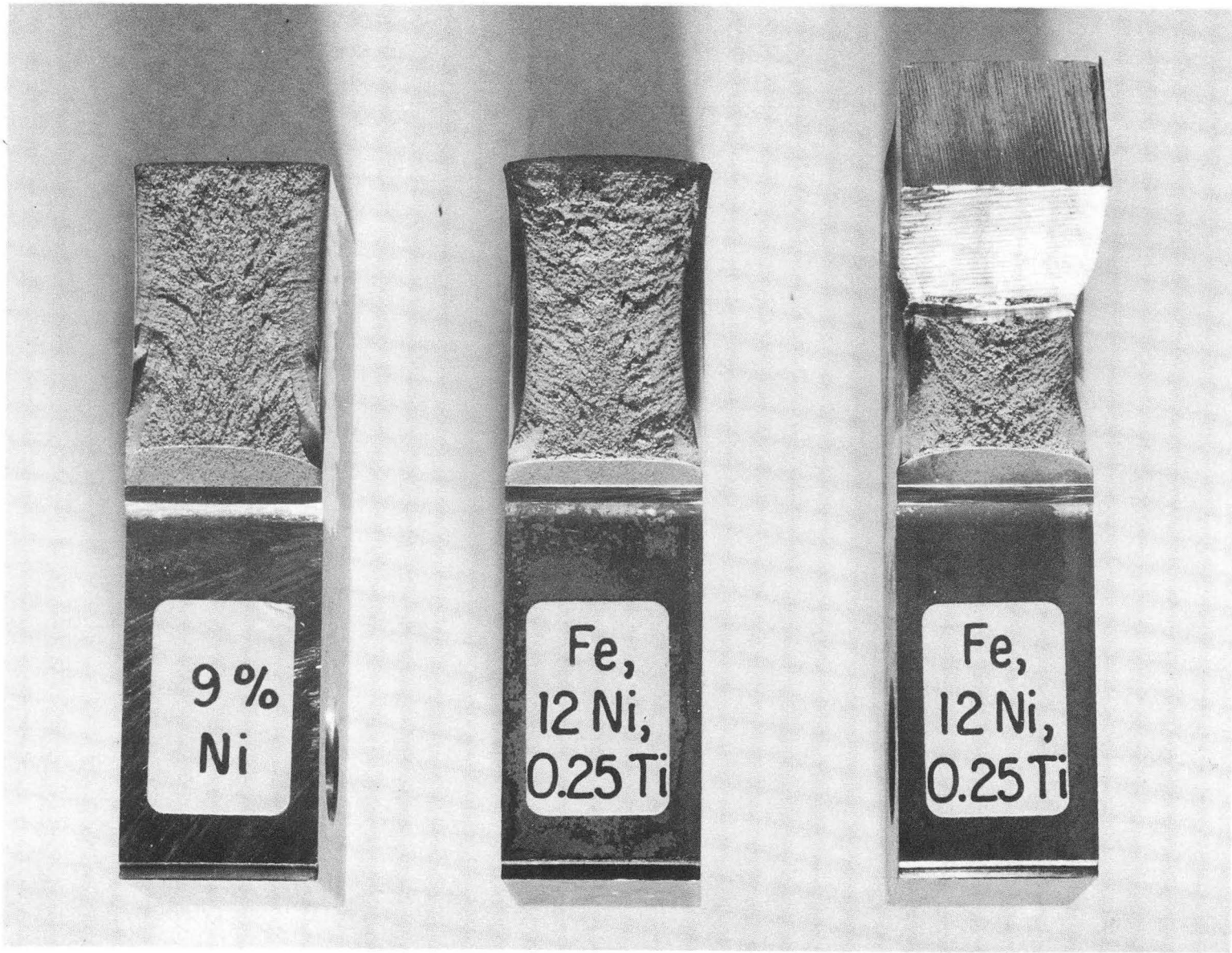
XBB 732-1215

Fig. 2



XBL 733-5855

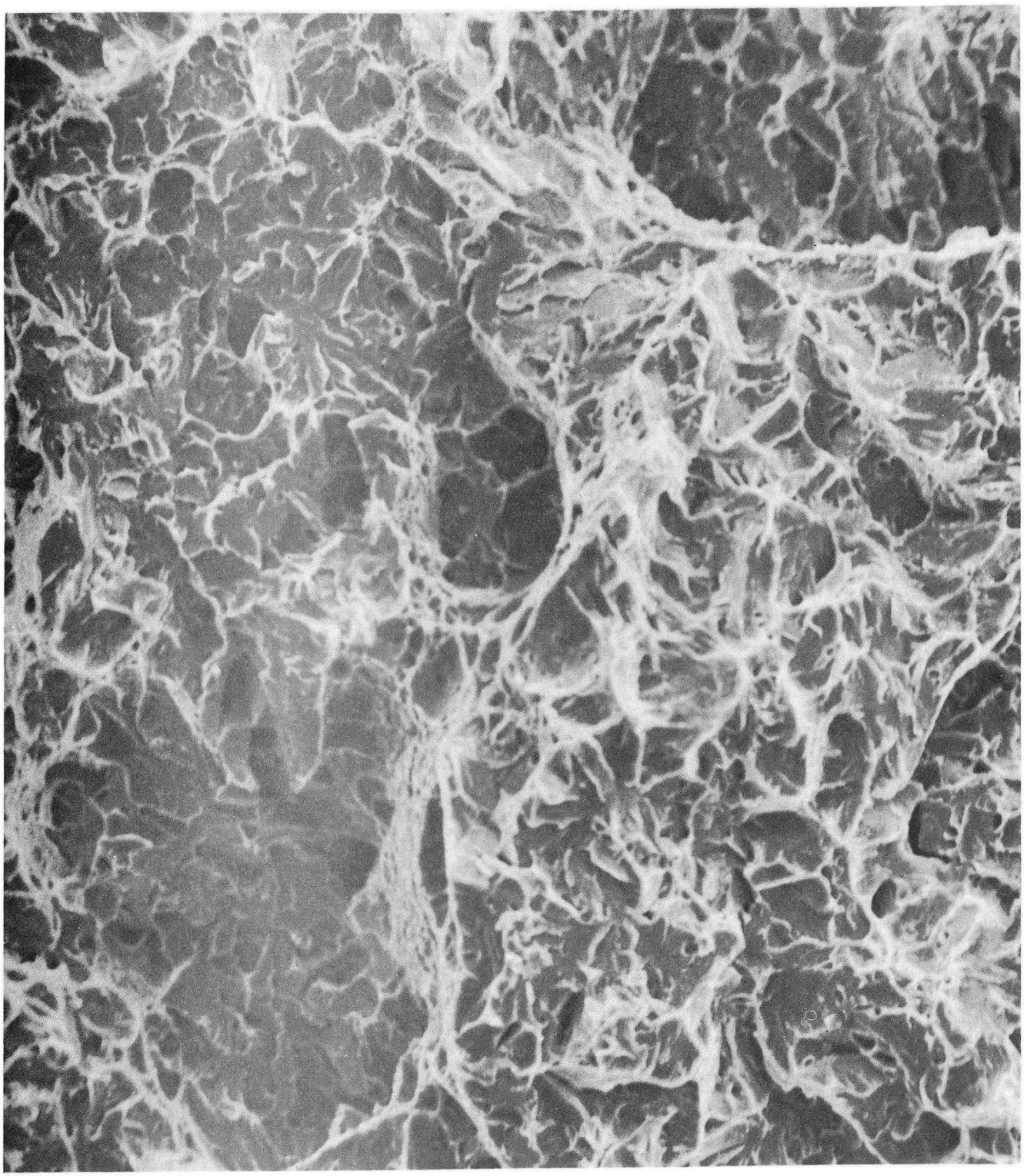
Fig. 3



(a) (b) (c) XBB 732-1220

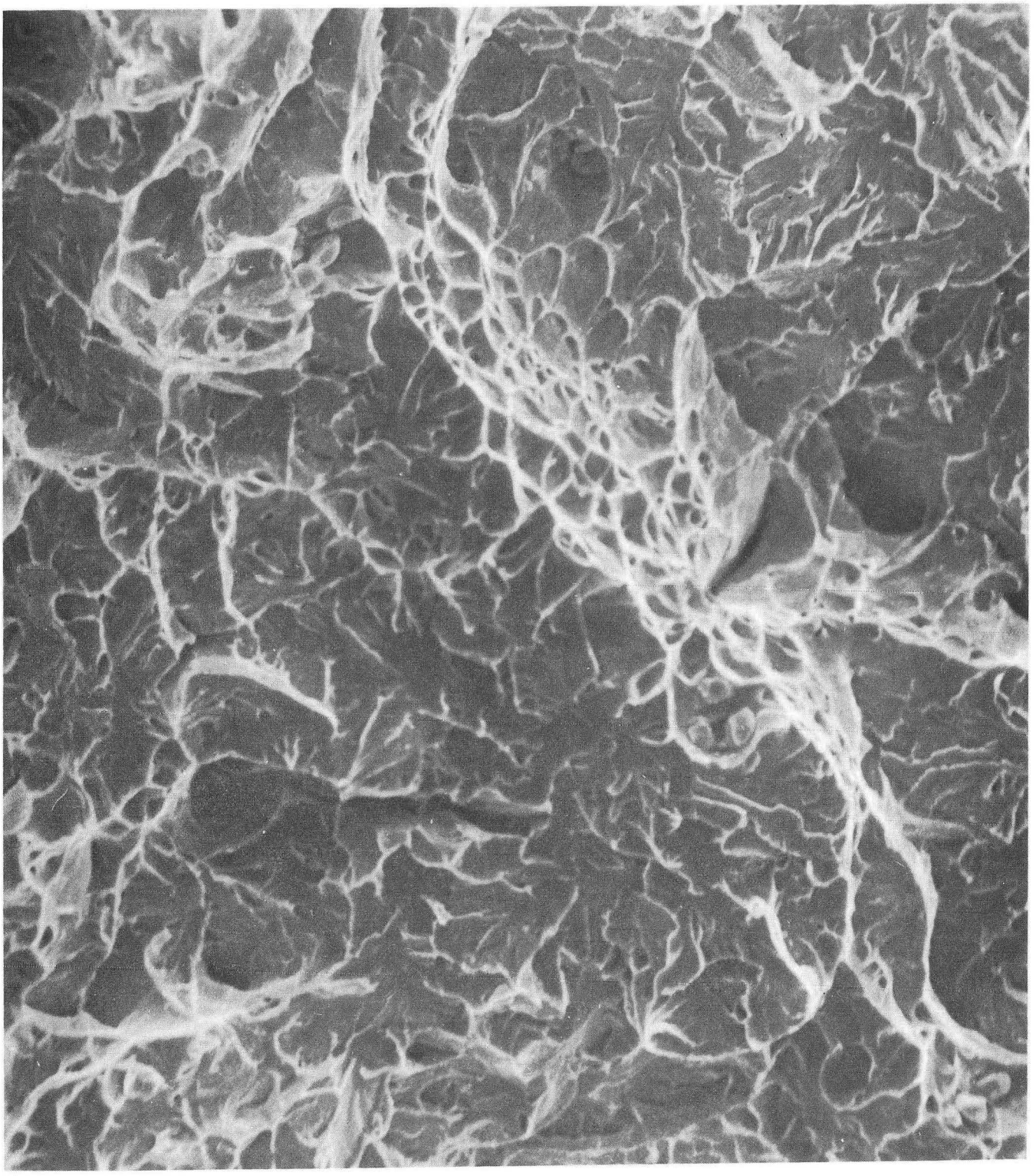
Fig. 4

0 0 0 0 3 9 0 2 7 4 5



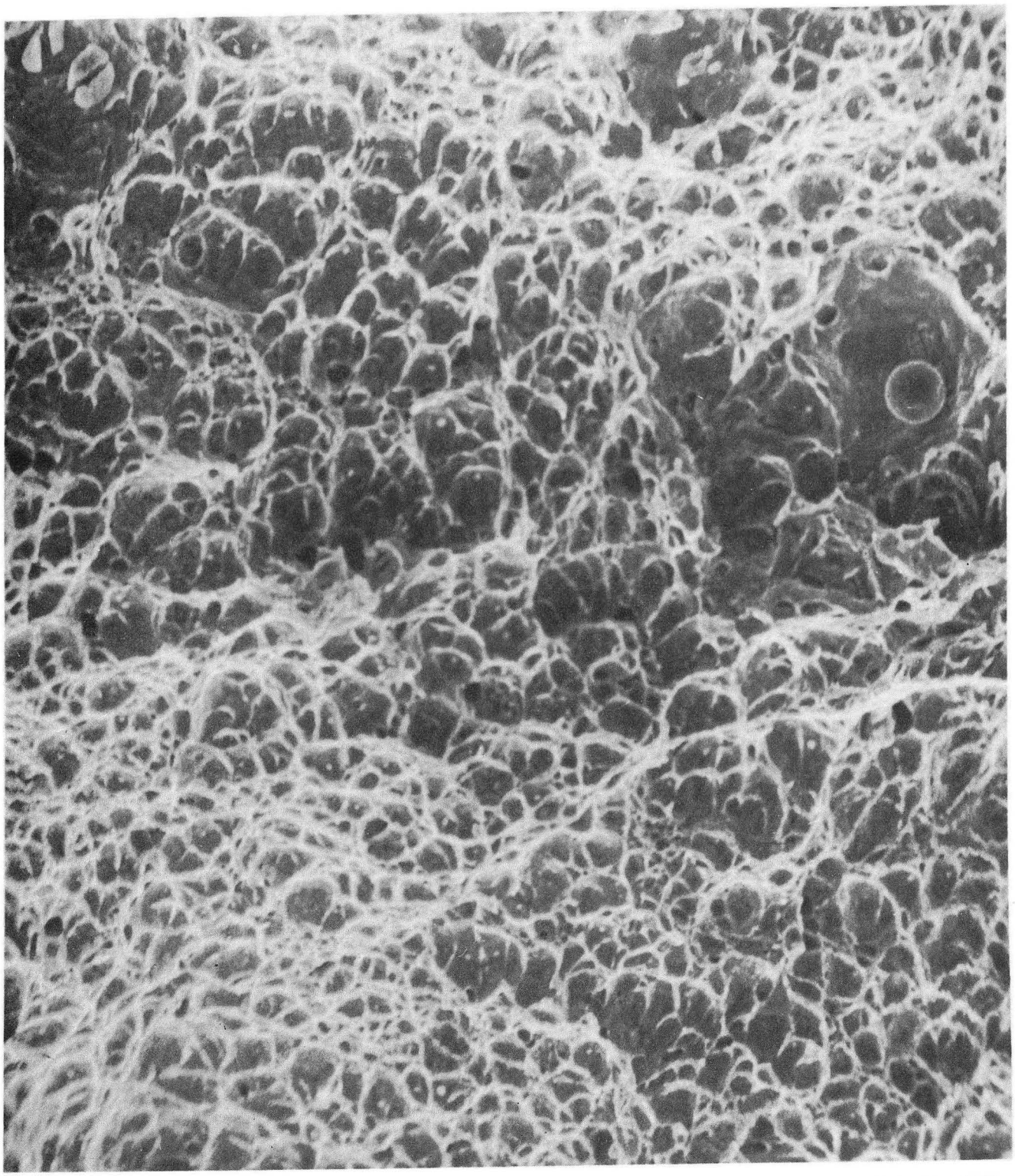
XBB 733-2165

Fig. 5a



XBB 733-2166

Fig. 5b



XBB 733-2167

Fig. 5c

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