CERN/EF/RF 87-7
3 December 1987

BRAZING OF NIOBIUM TO STAINLESS STEEL FOR UHV APPLICATIONS IN SUPERCONDUCTING CAVITIES

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ABSTRACT

At CERN a study has been made for brazing niobium to stainless steel AISI 316 LN [1] for use in large scale applications of superconducting cavities in accelerators. Results of metallographic studies and of mechanical and cryogenic tests are presented.

1. INTRODUCTION

At present the application of a large number of s.c. Nb-cavities for the Large Electron-Positron (LEP) storage ring is considered. This will imply a considerable number of UHV-tight joint between niobium and stainless steel flanges which are submitted to cryogenic temperature.

For a large storage ring problems of reliability and economy are of outstanding importance. Therefore a study was started at CERN for adequate methods of joining niobium parts to stainless-steel flanges. In fig. 1 a four-cell niobium cavity is shown. The cavity will be surrounded by a He tank which will be welded to the different openings of the cavity. In fig. 2, some of the openings are shown in more detail. Each opening is equipped by a stainless steel UHV flange necessitating a vacuum tight joint between the flange and the Nb tube which has to be operated at He temperature and must withstand repeated temperature cyclings between 4.2 K and 300 K. In addition the flanges of the large openings (beam tubes) have to support vacuum forces of several tons. In the following note the brazing method adopted will be discussed and results will be presented.

2. BRAZING PROCEDURE

The method previously developed [2] for vacuum brazing ($P < 10^{-5}$ mbar) with an alloy of 80% Au-20% Cu with a melting point of 889°C and brazing temperature of 960°C (which is also the temperature for annealing stainless steels) produces joints which are leak tight but when subject to large forces are not very reliable. The diffusion of iron from stainless steel at the interface niobium produces fragile intermetallic compounds. Such brazings are also very costly because of the price of the brazing material.

Brazing under vacuum with pure copper (type OFHC) having a melting point of 1083°C produces a better quality joint at lower cost. A quality of stainless steel such as the AISI 316 LN retains sufficient mechanical strength even after being heated to this temperature. In order not to oxidize the niobium, the vacuum of the brazing furnace should contain as little water vapour as possible.

The process shown in fig. 3 has been used to braze stainless steel flanges to niobium tubes which equip Nb superconducting cavities [3].

Before brazing, the stainless steel flanges have to be adjusted to the external diameter of the Nb tube, which are prepared by rolling and longitudinal welding. The inner diameter of the Nb tubes has to be adjusted with a stainless steel ring in order to compensate the difference in dilatation between the stainless steel and the Nb during brazing. After brazing a shear test of the brazed joint is performed by applying a force of 3500 KG to the joint followed by a leak test. Three successive immersions in liquid N_2 each followed by a warm-up to ambient temperature and leak test completed the procedure.

3. EXAMINATION OF JOINTS

The metallography of a series of copper brazed stainless steel to niobium joints has been carried out on samples taken from three 253 mm diameter brazed components. In order to assess the reliability of such a joint 20 samples from each component have been studied.

4. STUDY OF THE BRAZING WIRE

A 1 mm diameter oxygen free high conductibility (type OFHC) copper wire has been chosen as brazing material.

The chemical analysis yields the following values:

- Oxygen:	3 ppm	Antimony	
- Hydrogen:	1 ppm	Tin	
- Phosphor:	7 ppm	Manganese	< 40 ppm
- Sulphur:	18 ppm	Tellurium	
		Bismuth	

The high sulphur content (to be expected in an oxygen free copper) can favour oxygen penetration which reduces considerably the tensile strength. However, brazing under vacuum eliminates this drawback as confirmed by micrographic examination.

An examination of the wire proves that the lubricants, generally of organic origin, used in the wire drawing operation, have been properly removed from the wire surface. If such oils remain on the surface they turn to carbon when the brazing material melts and migrate to the stainless steel/brazing/niobium interfaces thus making the joint more fragile. A search for inclusions confirmed the high purity of the copper. Furthermore no trace of incrusted foreign bodies due to the drawing operations has been found.

5. MICROGRAPHIC EXAMINATION

The external appearance of the three brazed components vary from one to the other. One, for example, shows an excess of brazing copper spread onto the stainless steel flange.

The samples were taken from potentially critical zones: on the bolt hole axis, at the longitudinal weld of the niobium sleeve, in the zones of irregular or scant brazing material deposit (fig. 4). The purpose of these examinations are:

- To detect defects such as cracks, loss of cohesion, shearing, stress cracking, voids.
- To check that the melted metal has fully wetted the mating surfaces and that the space between the latter is completely filled after brazing.
- To check that the brazing material has run satisfactorily over the two metals to be joined, i.e. it has dissolved part of the metal to form an alloy. This property determines the distances to which the brazing material moves from its original position.

- To estimate the diffusion which, in this case, can be considered as a thermally activated diffusion forced by thermal activation modified by the presence of external forces (stress induced by the difference in thermal expansions of different materials).

This is not a physical examination to determine the displacement coefficients, the activation energies, the frequency factors but merely a check on the evolution of materials as a function of time and of temperature. This examination aims at describing the diffusion of impurities, verify the evolution of "short-circuits" in the transport of materials involving large size defects such as the grain boundaries. The results of the analysis of the three brazed components can be summed up as follows:

- On every sample the brazing material had melted correctly and completely filled the free space between the different parts of the component (fig. 6).
- The systematic search for typical defects such as cracks, etc. showed high quality joints; the rare voids found were always located in the grooves housing the copper wire (fig. 7).
- The thickness and the shape of the melted layer varies a lot from one component to the other (fig. 7). It seems that the thickness of the melted metal layer is indirectly proportional to the clearance between components.
- In every case, the copper has "run" correctly i.e. it has combined well with the stainless steel by absorbing a small quantity of iron to form an iron rich compound (stainless steel compound) on the niobium side. The dissolution process is not very important on the niobium face. The thickness of the compound varies a lot and develops with the increase of the clearance between mating components.
- The study of the diffusion shows in every case a strong development of the copper in the stainless steel grain boundaries (short-circuits).

It has not been possible to distinguish a difference between the 3 components. The most affected zones are those neighbouring the grooves housing the copper wire (fig. 8). The segregation of impurities in the grain boundaries has not been noted. On the niobium sleeve side, the diffusion is very small and the analysis through energy dispersive X-ray analysis confirms the presence of an iron rich film (compound) at the niobium-stainless steel interface (fig. 9).

6. FRACTURE EXAMINATION

In order to analyze more precisely the characteristics of this joint, samples of the three components have been fractured at ambient temperature. In all cases, the results are identical: the rupture always occurs within the "compound" at the niobium-copper interface (fig. 10). This rupture is of the brittle type through cleavage. This mode of rupture occurs when the stress locally exceeds the cohesion of the metal. It leads to the rupture of the interatomic bonds. The rupture plane is made up of a juxtaposition of bright and flat facets. There is no trace of even small plastic deformation (figs 11 and 12).

The obvious presence of a network of well-defined lines running along the structural defects as well as that of "tangues" corresponding to local decohesion always reveal a fracture without plastic deformation (fig. 12). This brittleness is confirmed by the tilting effect observed during the fracture which corresponds to the shearing of twin generally associated with ruptures through cleavage.

7. CONCLUSION

The comparative examination of these assemblies of the stainless steel to niobium joints achieved by copper brazing shows:

- No difference between components subject to liquid nitrogen cooling and mechanical strain and those not cycled.
- Little influence of varying surface finishes (machining) of the mated components (slight differences in the molten layer) on the joint.
- No major defects like cracks, etc.
- A complete filling of the free space by the brazing material.
- Good braze flow in particular on the stainless steel limited diffusion with a little infiltration in the stainless steel grain boundaries.
- A certain brittleness when a plastic deformation is applied, due to the presence of an iron rich compound located at the niobium copper interface caused by an excessive dissolution of the stainless steel.

Finally, this joint satisfies all the criteria of a brazing to be used in ultra-high vacuum work, in spite of slight mechanical brittleness.

Acknowledgments

We would like to thank S. Neil, H. Lengeler and R. Stierlin for their help during the course of preparing this paper. We are especially grateful to T. Wikberg for his valuable support of the work reported in this paper.

Our thanks go also to C. Ruivet and R. Jacob for their contribution to the success of this development work.

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[1] CERN Technical Specification No. 66, Rev. 6.

[2] J. Susta, Proc. of the 2nd Workshop of RF superconductivity, CERN, Ed. H. Lengeler, (1984) 597.

[3] G. Arnolds-Meyer et al., CERN/EF 87-9.

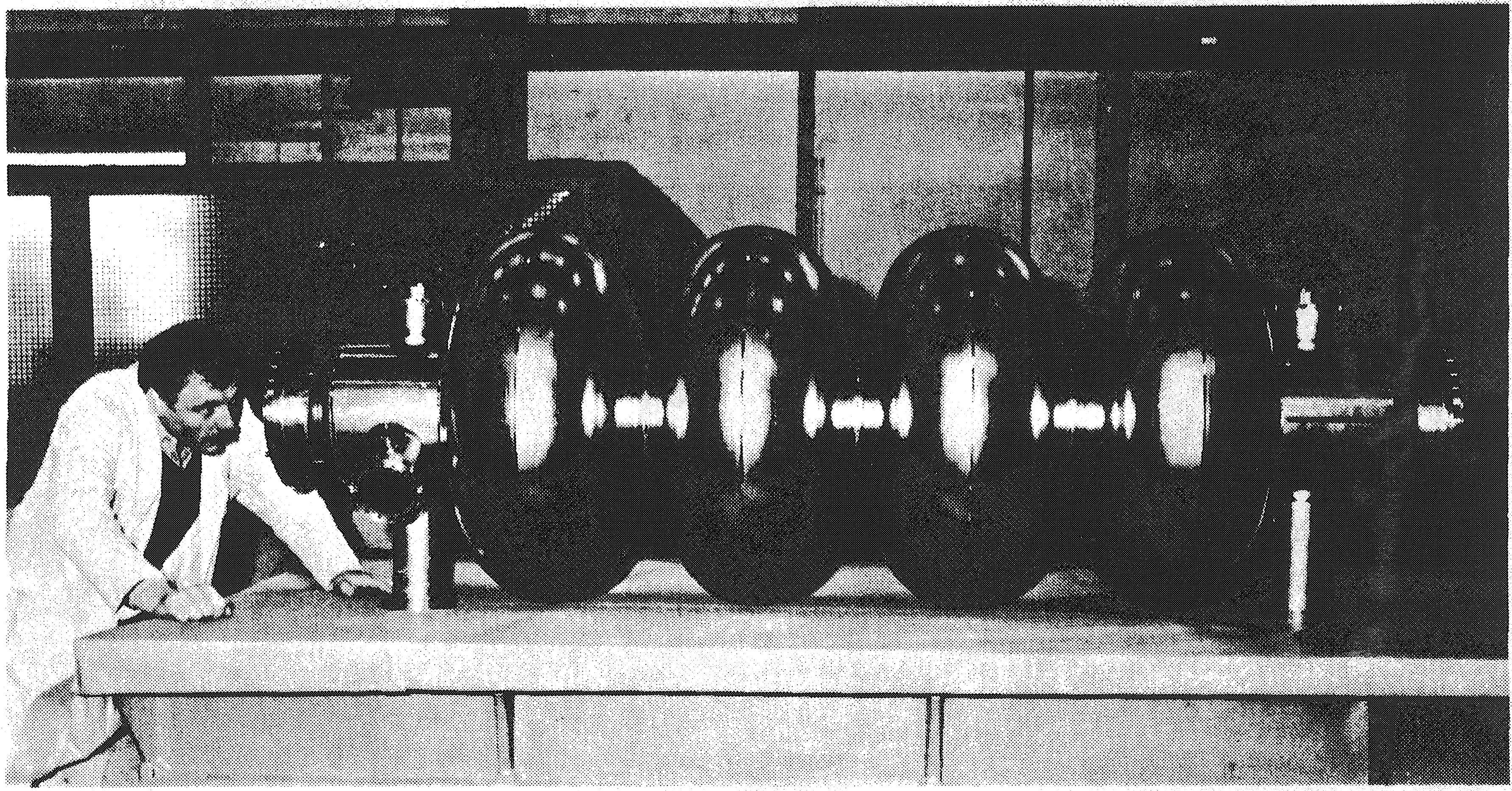


Fig.1

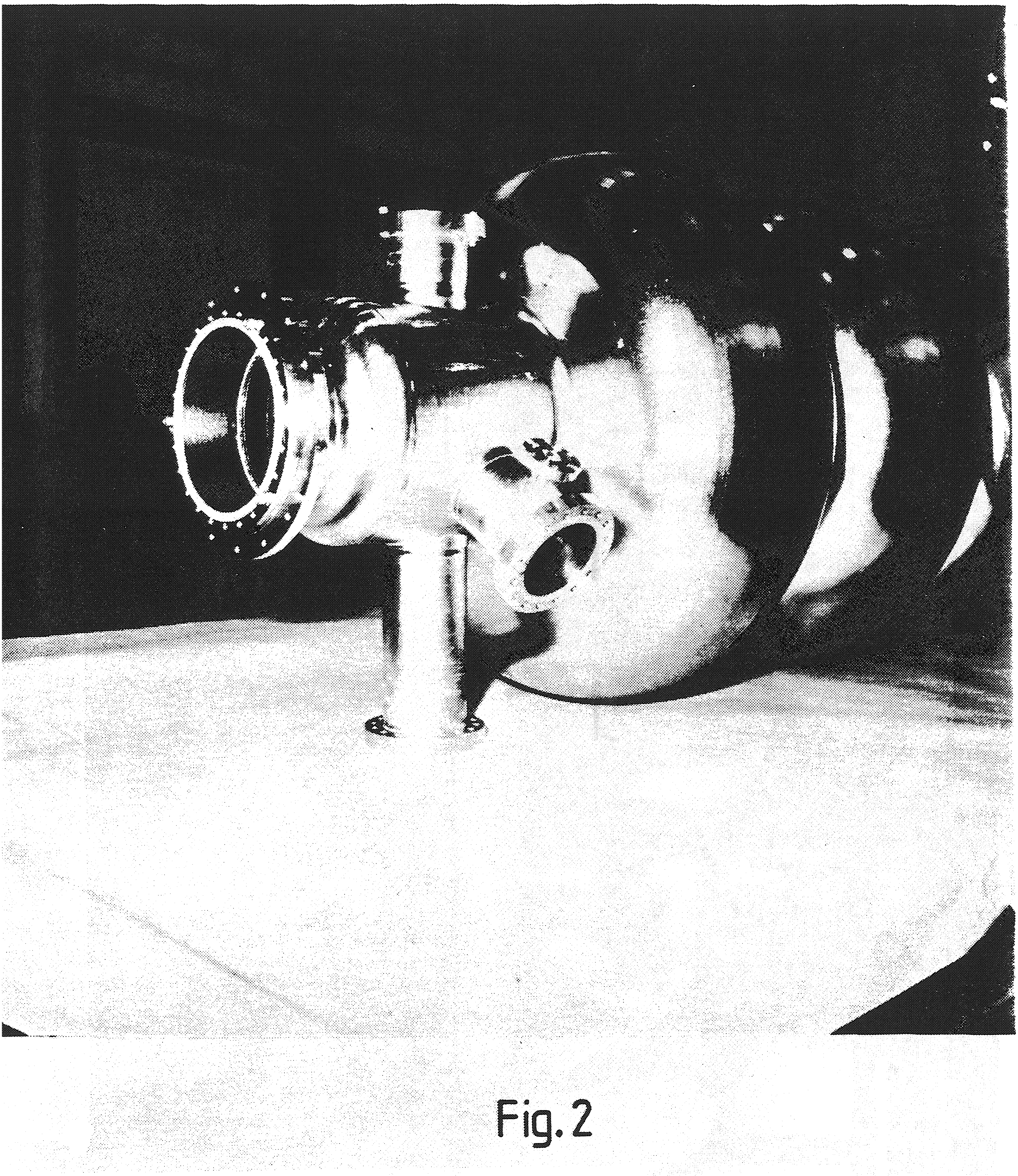
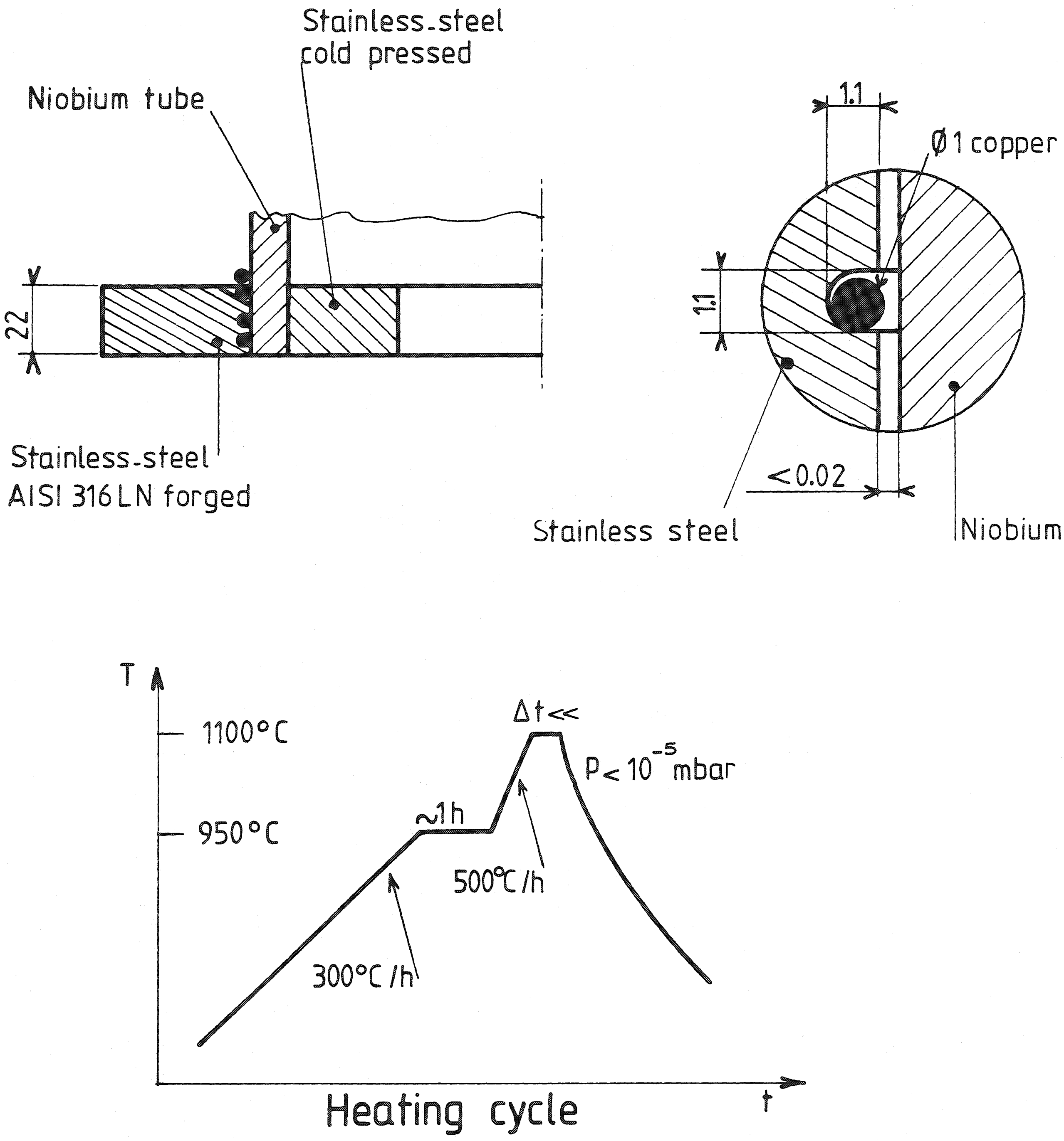


Fig. 2



Stainless-steel
cold pressed
Niobium tube
22
Stainless-steel
AISI 316 LN forged
1.1
Ø1 copper
1.1
<0.02
Stainless steel
Niobium
T
1100°C
950°C
Δt<<
P< 10^-5 mbar
~1h
500°C/h
300°C/h
t
Heating cycle

Fig. 3

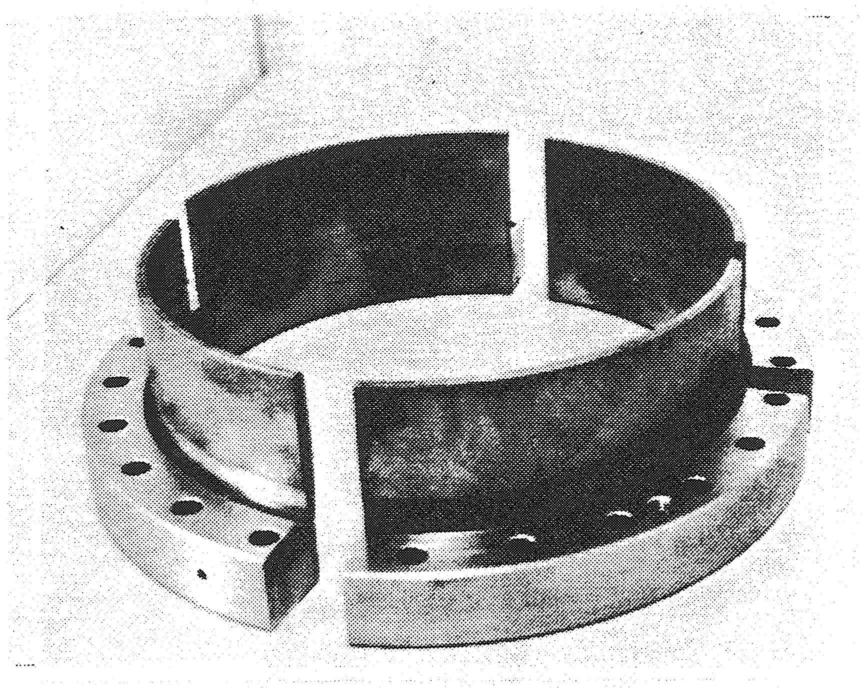


Fig.4

Position of metallographic specimens for Nb stainless steel joint, ext. Ø 253 mm.

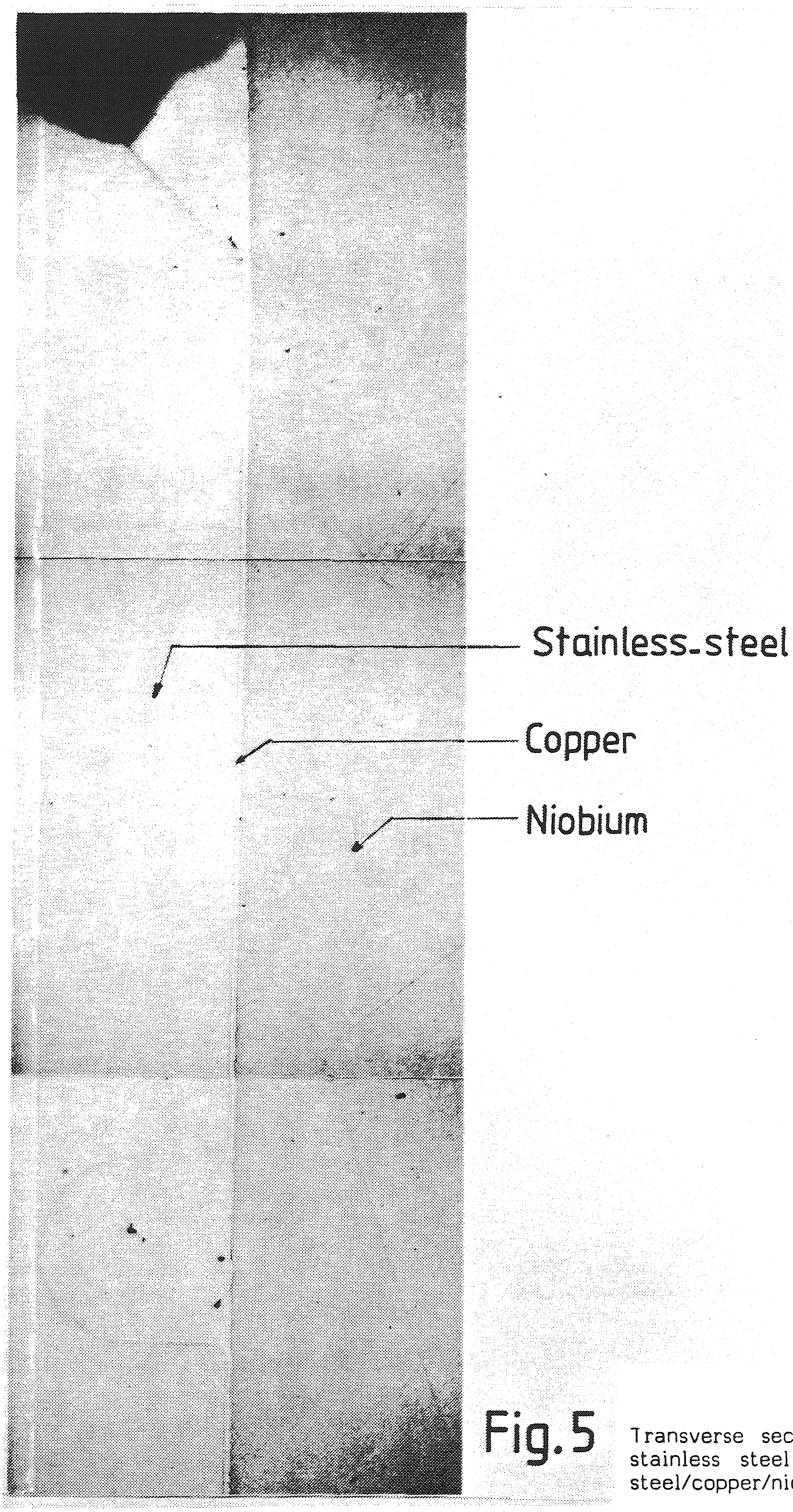


Fig. 5 Transverse section of niobium stainless steel braze stainless steel/copper/niobium.

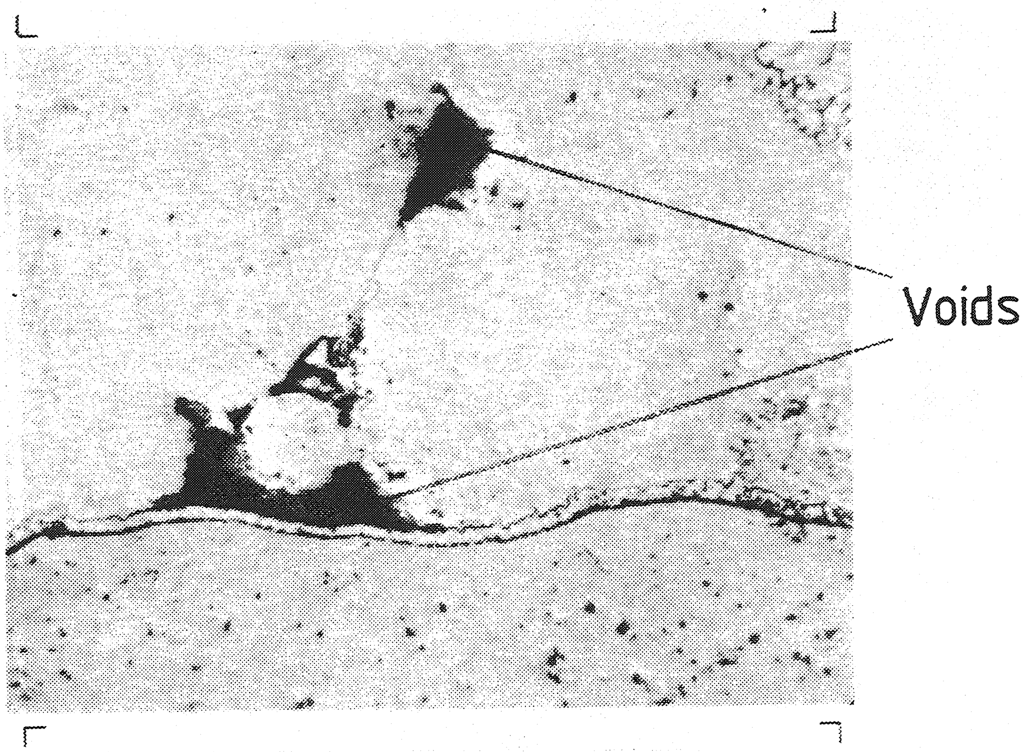
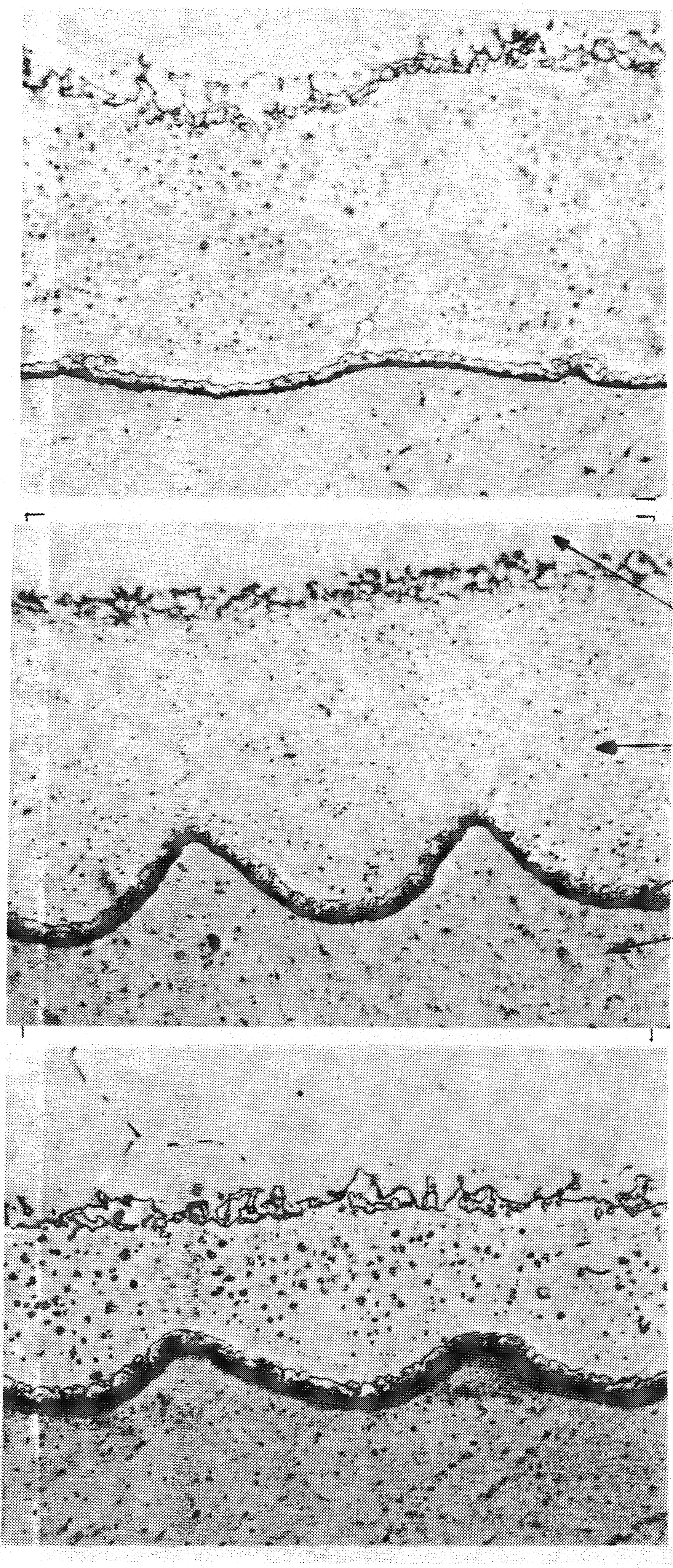


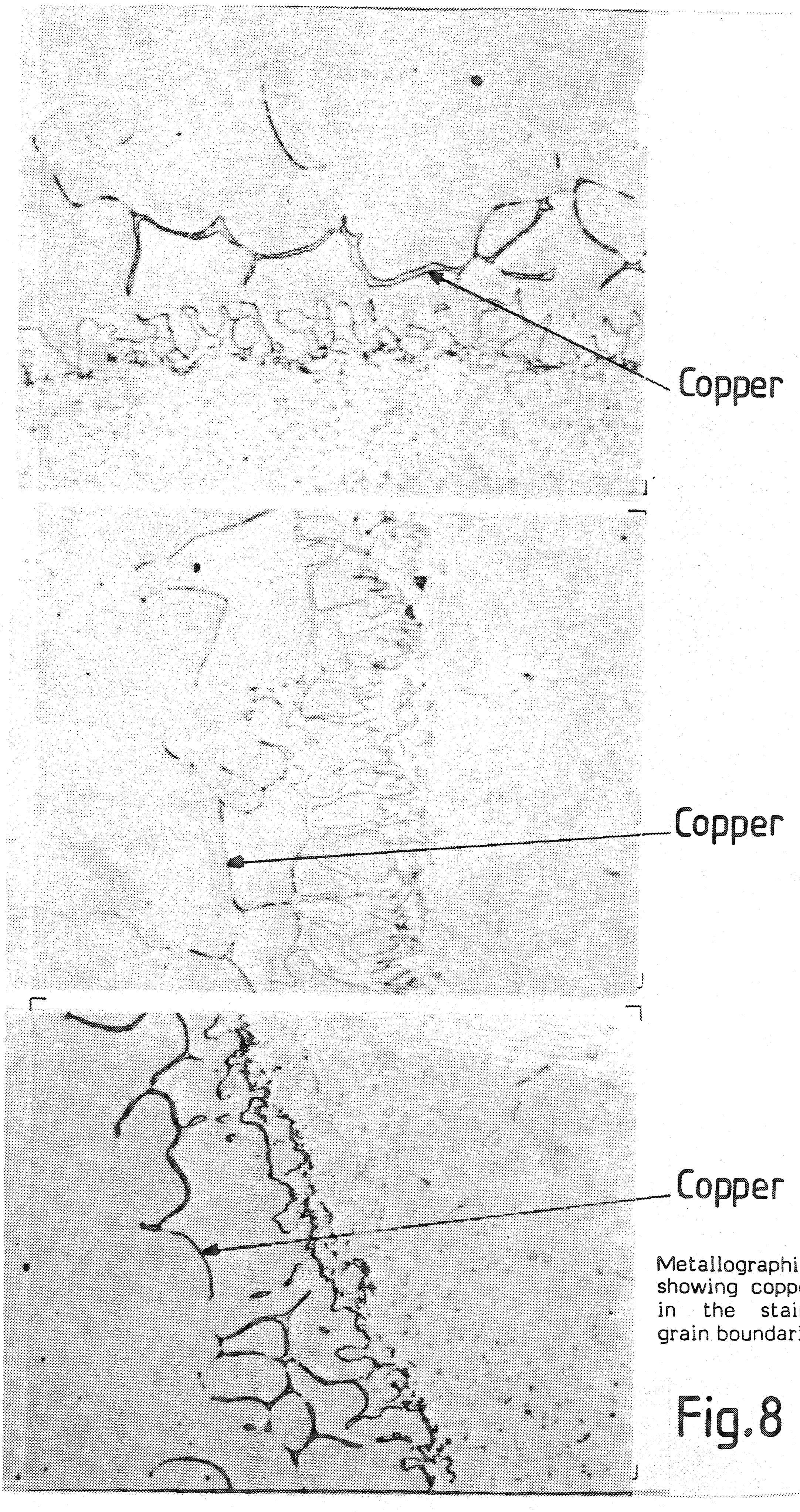
Fig. 6

Metallographic section showing rare voids.



Metallographic section showing in detail the brazing region.

Fig.7



Metallographic section showing copper diffusion in the stainless steel grain boundaries.

Fig.8

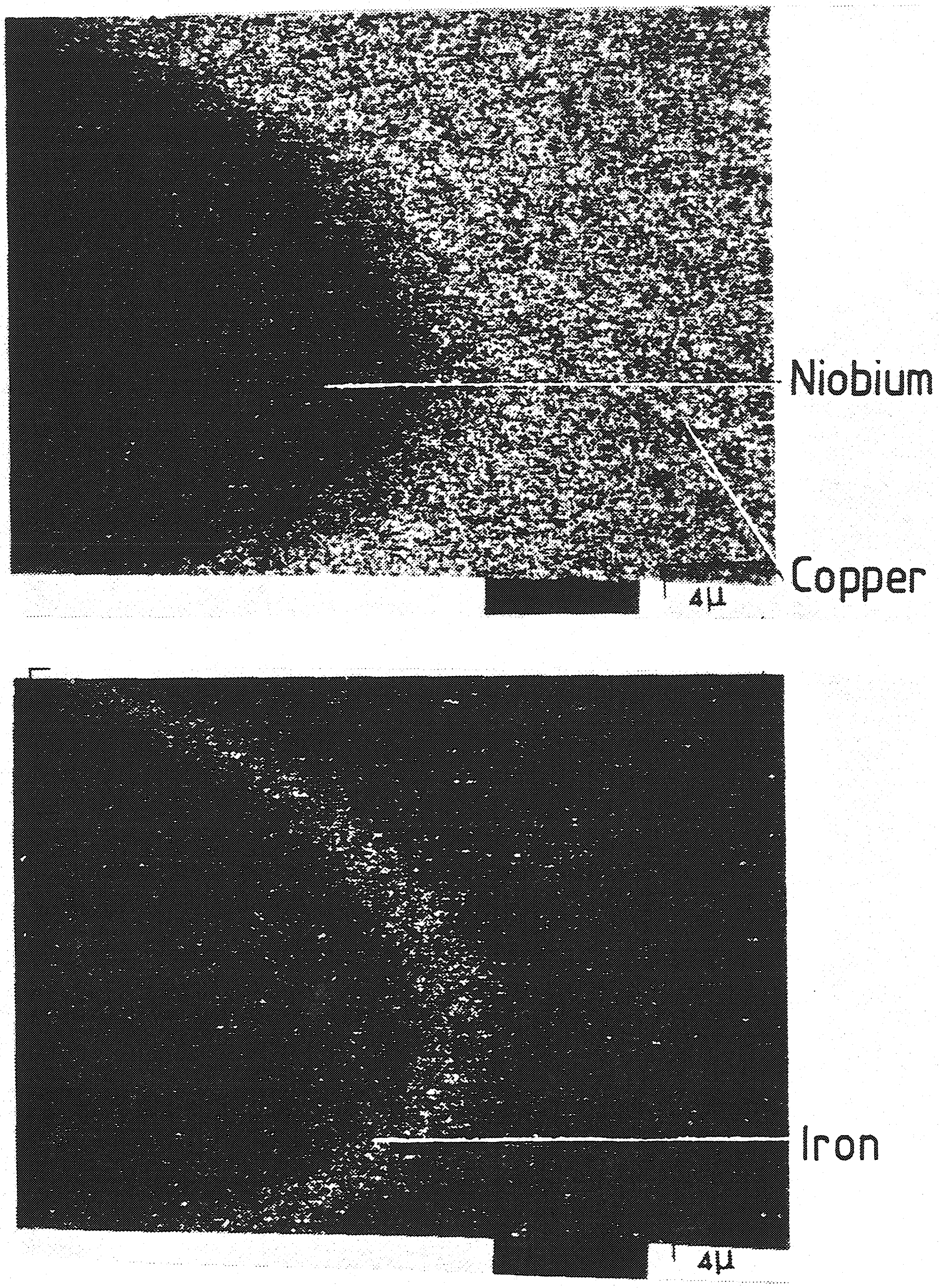


Fig.9

Iron distribution on the Nb side as determined by energy dispersive X-ray analysis.

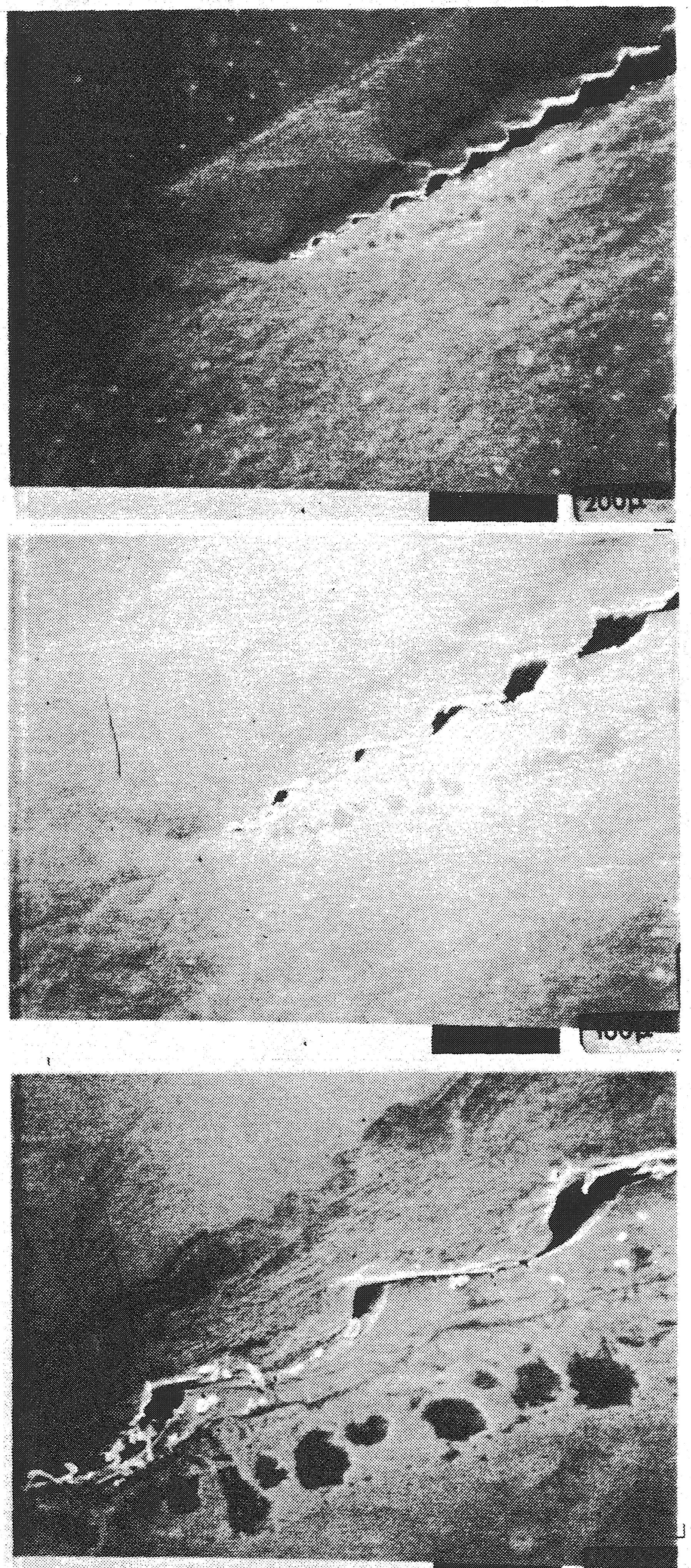
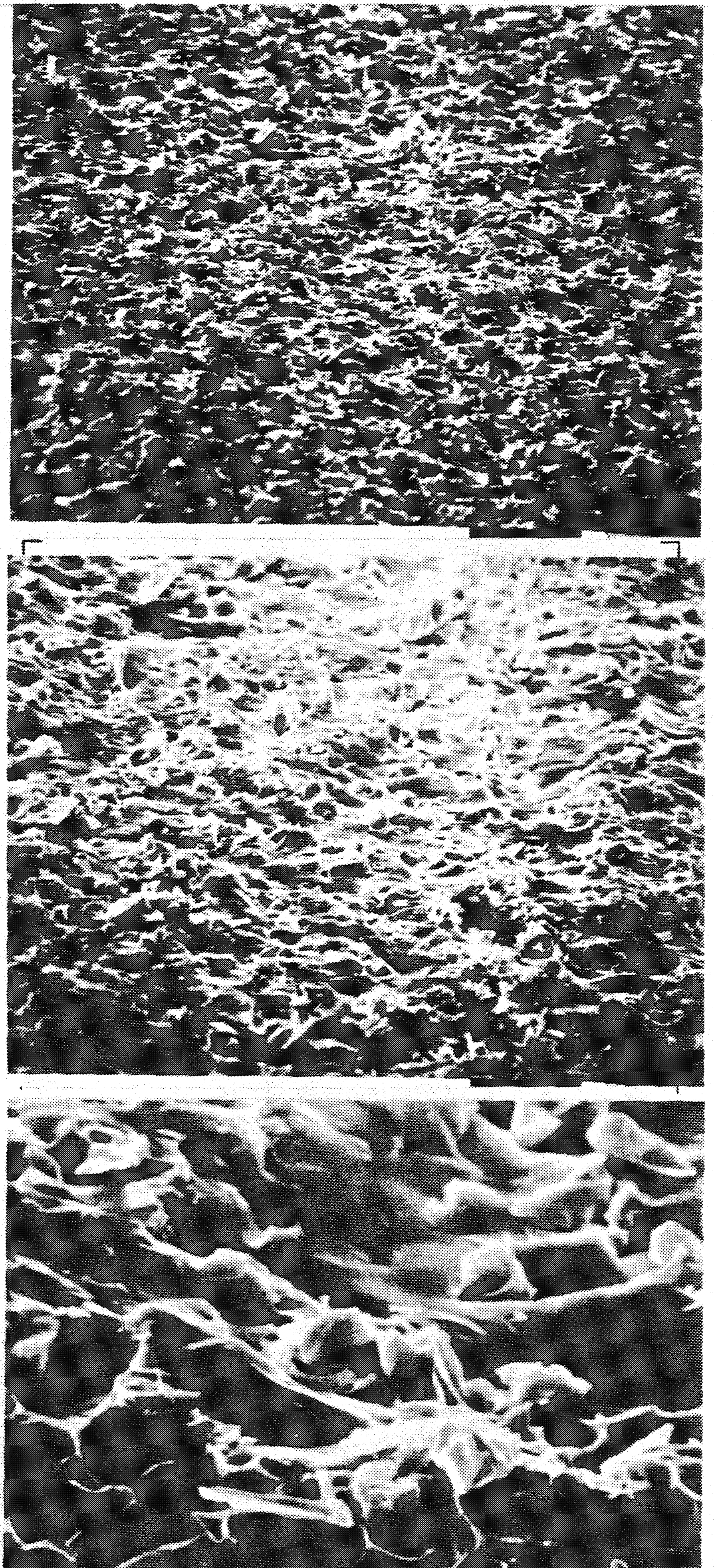


Fig.10



Fracture examination with electronic microscope at stainless steel flange side.

Fig.11

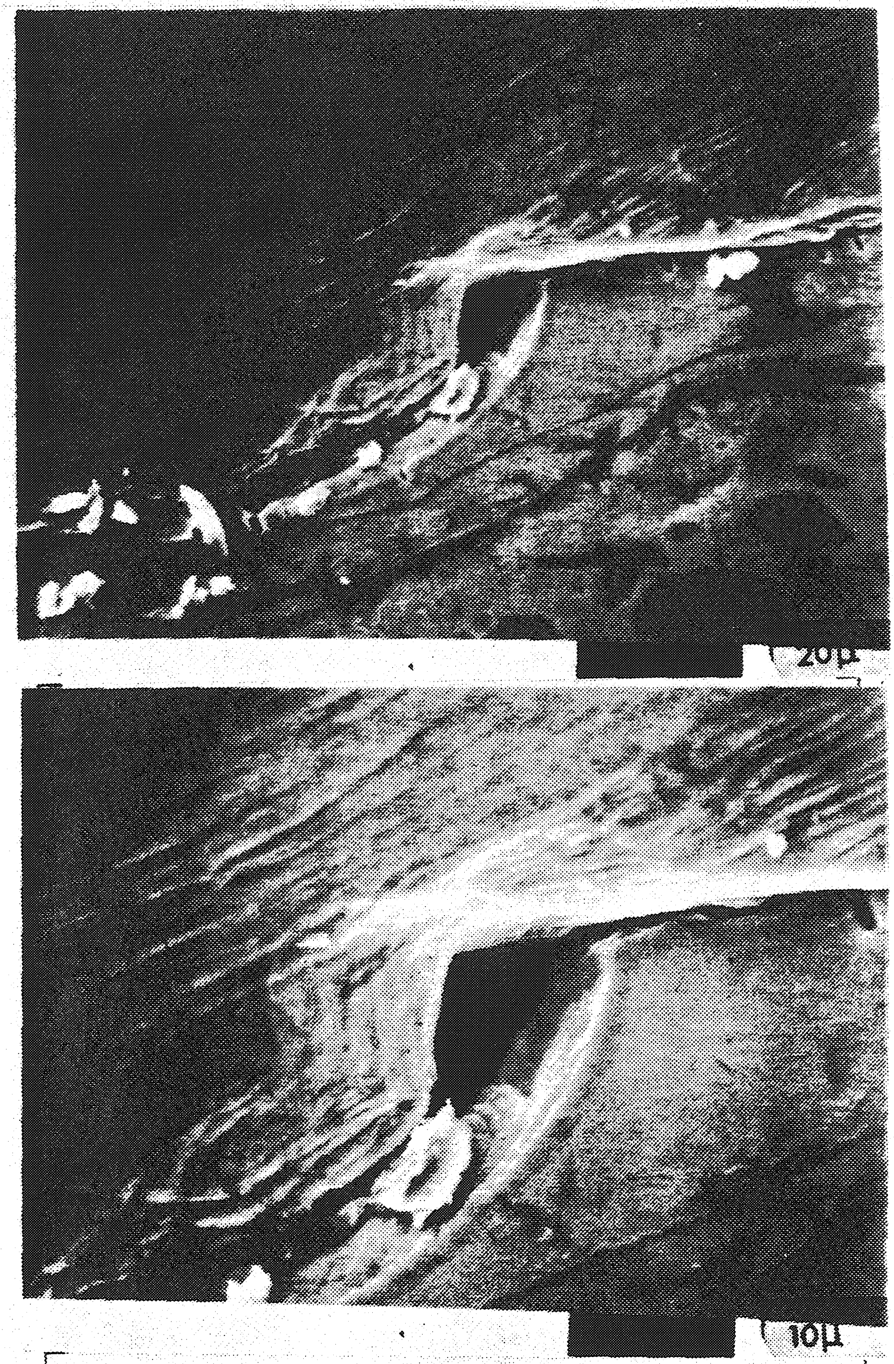
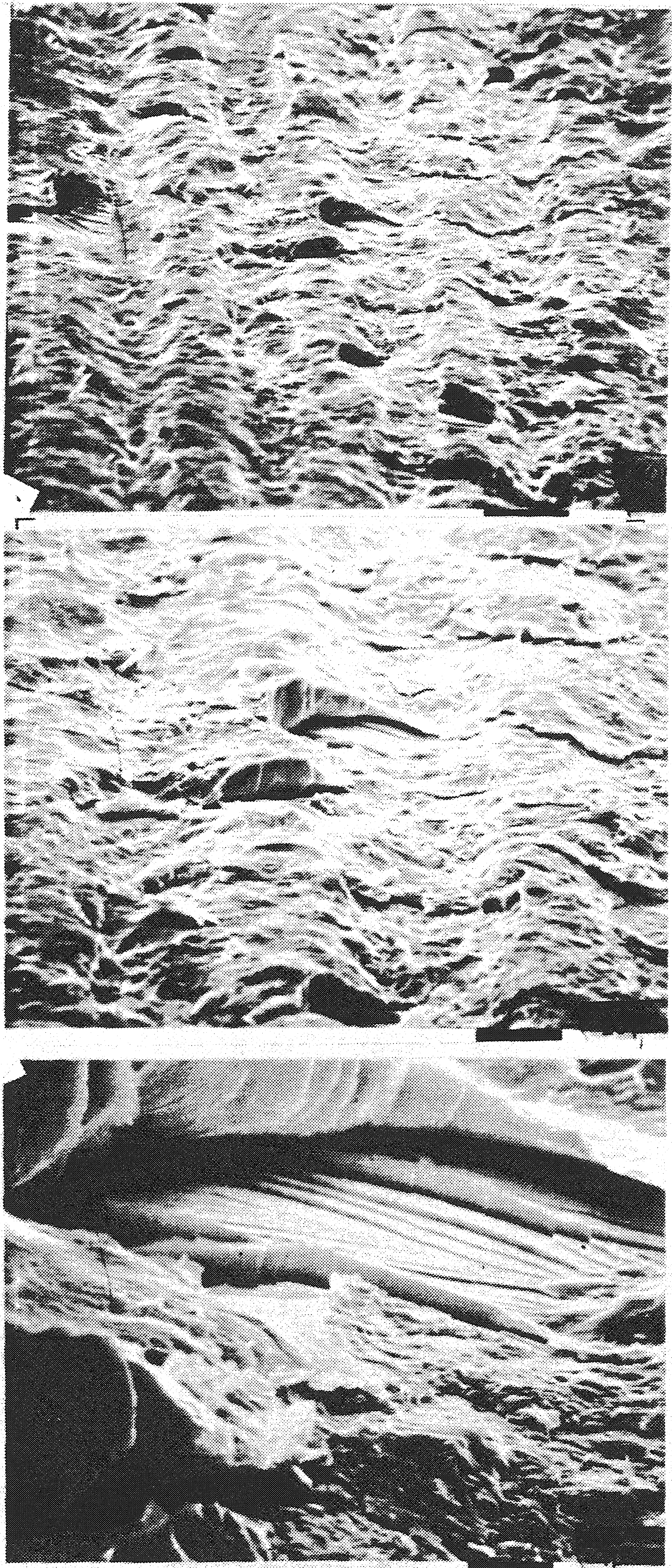


Fig.10 Cont'd

Progression of crack within the "compound" at Nb/stainless steel interface.



Fracture examination with electronic microscope at Nb side (brittle crack due to cleavage).

Fig.12