1/5/78

LA-7019-MS
Informal Report

UC-25
Issued: November 1977

Examination of Copper or Nickel Coats on W-Base Composites

E. G. Zukas

los alamos
scientific laboratory
of the University of California
LOS ALAMOS, NEW MEXICO 87545

An Affirmative Action/Equal Opportunity Employer

UNITED STATES
DEPARTMENT OF ENERGY
CONTRACT W-7405-ENG. 36

EXAMINATION OF COPPER OR NICKEL COATS ON W-BASE COMPOSITES

by

E. G. Zukas

ABSTRACT

Tungsten-base composites coated with either nickel or copper were heat-treated at elevated temperatures to produce a diffusion bond between the composite and the coat. At temperatures to 473 K, no diffusion bond was achieved and there was no improvement in low-temperature ductility. At temperatures above 920 K, the coat and base bonded and the ductility was improved. Microprobe analysis confirmed that a diffusion bond formed.

I. INTRODUCTION

A process for improving the low-temperature ductility of tungsten-base composites by coating with a ductile metal followed by heat-treatment has been reported.[1,2] For coated specimens heat-treated at low temperatures (473 K) where a diffusion bond between the coat and the base composite was unlikely, there was no discernable improvement in ductility over that of the same composite in the uncoated condition. However, at higher heat-treating temperatures, where the coat and base could become integral by codiffusion, improved ductility was achieved. As stated earlier,[1,2] nondiffusion bonded coatings acted merely as envelopes and had no effect on the base, whereas bonded coatings served as a ductile surface for the base, and reduced points of stress concentration at spheroid junctions and at imperfections in the sintered composite. Had these conclusions been accepted without experimental verification, this report would not have been necessary.

Tested tungsten-base composite specimens were examined. The ductile specimens were those in which diffusion bonding occurred during the heat-treatment.

II. EXPERIMENTAL TECHNIQUE

The three different heat-treating conditions studied after coating were a) the copper-coated 94.75% W-3.5% Ni-1.5% Fe-0.25% Re after 1 h at 473 K, b) the copper-coated 95% W-3.5% Ni-1.5% Fe after 1 h at 923 K, and c) the nickel-coated 94.5% W-3.5% Ni-1.5% Fe-0.5% Pt after 1 h at 1223 K. The specimen heat-treated at 473 K broke during the bend test, whereas the other two passed through the bend test fixture without fracturing. These specimens were sectioned longitudinally for examination. Microprobe examination was used to determine diffusion behavior.

III. EXPERIMENTAL RESULTS

A nonscientific test was used to examine coat adherence. A scalpel was used in an attempt to separate the coat from the composite base. Patches of coat were readily removed from the copper-coated composite which had been heat-treated at 473 K, showing essentially no coat-base diffusion, and these specimens were eliminated from further study. The coatings on the other specimens were adherent and could not be removed mechanically.

Longitudinal sections including the bend were then mounted in epoxy. The specimens were ground through 600-grit SiC-coated paper using water as a lubricant, followed by approximately 70 minutes on an Automet polisher using 1-μm diamond with ethlene glycol. The big difference in hardness between the tungsten spheroids and the annealed copper or nickel coats made it impossible to get rid of the polishing relief. Thus, all photomicrographs are of the specimens in the as-polished condition. Typical photomicrographs of the copper-coated composite are shown in Figs. 1 and 2. There are a number of pores in the copper, and a demarcation line about 70 μm from the coat surface indicating that perhaps the coating process was stopped and restarted (perhaps after checking coat thickness). Surprisingly, this boundary remains after heat-treatment at 923 K, whereas the boundary between the copper coat and composite matrix is not readily apparent. The base composite contains a few pores, but generally appears to be reasonably sound. However, as reported earlier,[1] the bend ductility angle was only 14° for the uncoated specimen, whereas the coated specimen passed through the bend-test fixture without fracturing. Specimens coated with nickel, and heat-treated for 1 h at 1223 K are shown in Figs. 3 and 4. In these specimens, 0.5% Pt was substituted for 0.5% W. These specimens contained quite a bit of porosity, with many pores on the surfaces after the specimens were machined and polished. The bend ductility angle for the uncoated material was 16°, not significantly different from the relatively pore-free material. The heat-treated specimens passed through the bend-test fixture without fracturing. The nickel coat, which appears to have alloyed with the composite matrix, spans the gaps at the pores, thereby decreasing their effectiveness as stress raisers and reducing their tendency to promote fracture.

A number of microprobe scans were run to determine the change in chemical composition as a function of position across the coat-base matrix interface. In this analysis, the composition at each position is determined by counting for 10 s and comparing this output with that for the pure element. Scans consist of such countings at 2-μm intervals over the entire scan path. The output may be affected slightly because of differences in surface elevation for the different components in the structure, such as relief produced by mechanical polishing or because of selective attack during etching. For these composites, the matrix is softer than the spheroids, which causes relief during polishing, and etching selectively attacks the matrix. These specimens were analyzed in the as-polished condition to keep such effects at a minimum.

Eight individual microprobe scans for the 95% W-35.% Ni-1.5% Fe composite coated with copper followed by heat-treating in vacuum for 1 h at 923 K are shown in Figs. 5 through 8. Arrows are used to denote the scan path but do not define the starting point. Since the spheroids are essentially pure W, the scan paths can be matched to position in the structure. The interdiffusion appears to be between the nickel in the matrix and the copper coat. The width of the alloy band is about 6 μm, in fair agreement with what should be expected based on the diffusivity for the times and temperatures used. The important point here is that there is chemical bonding between the copper coat and the base composite.

Eight individual microprobe scans for the 94.5% W-3.5% Ni-1.5% Fe-0.5% Pt composite coated with nickel followed by heat-treating in vacuum for 1 h at 1223 K are shown in Figs. 9 through 12. The analytical results show that there is diffusion between the nickel coat and the base composite. In some cases, the diffusion layer seems to be quite wide, but this should be expected in view of the relatively high heat-treatment temperature. However, there is one aspect of the results which may not appear reasonable. In some instances, W diffuses readily into the nickel coat, whereas at other locations, it does not. The same situation occurs with Fe. This behavior is probably similar to that observed in the activated sintering of W with Ni where very rapid diffusion takes place along grain boundaries and along certain crystallographic planes.[3,4]

The microprobe results show definitely that a diffusion bond is formed between the coat and the base. Further analysis of the results shows that the matrix composition is not completely homogeneous throughout the composite. This is rather surprising in view of the careful mixing of the powders before the W is added. However, there is the possibility that some type of separation occurs during sintering since W and Ni can alloy. From a practical standpoint, there may be no advantage in achieving complete homogeneity, but the effects should be investigated, perhaps by using prealloyed powder of the final matrix composition (including W).

IV. CONCLUSIONS

1. Ductile coatings on W-base composites which are not diffusion bonded have no effect on low-temperature ductility.
2. Ductile coatings on W-base composites which are heat-treated at high temperatures do diffusion bond and do promote low-temperature ductility.
3. The effect of stress raisers such as pores and spheroid junctions can be at least partially overcome by a diffusion-bonded ductile coat.
4. The matrix composition in liquid-phase sintered tungsten-base composites is not completely homogeneous.

ACKNOWLEDGMENTS

The author expresses his appreciation to Los Alamos Scientific Laboratory, Group CMB-6, for making the composites and electroplating the bend specimens and to Group CMB-1 for microprobe analysis.

REFERENCES

1. Eugene G. Zukas, "Process for Improving the Low Temperature Ductility of Tungsten-Base Composites," Los Alamos Scientific Laboratory report LA-5962-MS (May 1975).
2. Eugene G. Zukas, "Coating Tungsten Composites for Improved Low-Temperature Ductility," Met. Trans. B (1976) pp. 49-54.
3. G. H. Gessinger and H. F. Fischmeister, "A Modified Model for the Sintering of Tungsten with Nickel Additions," J. Less-Common Metals 27 (1972) pp. 129-141.
4. I. Amato, "On the Mechanism of Activated Sintering of Tungsten Powders," Mater. Sci. Eng. 10 (1972) pp. 15-22.

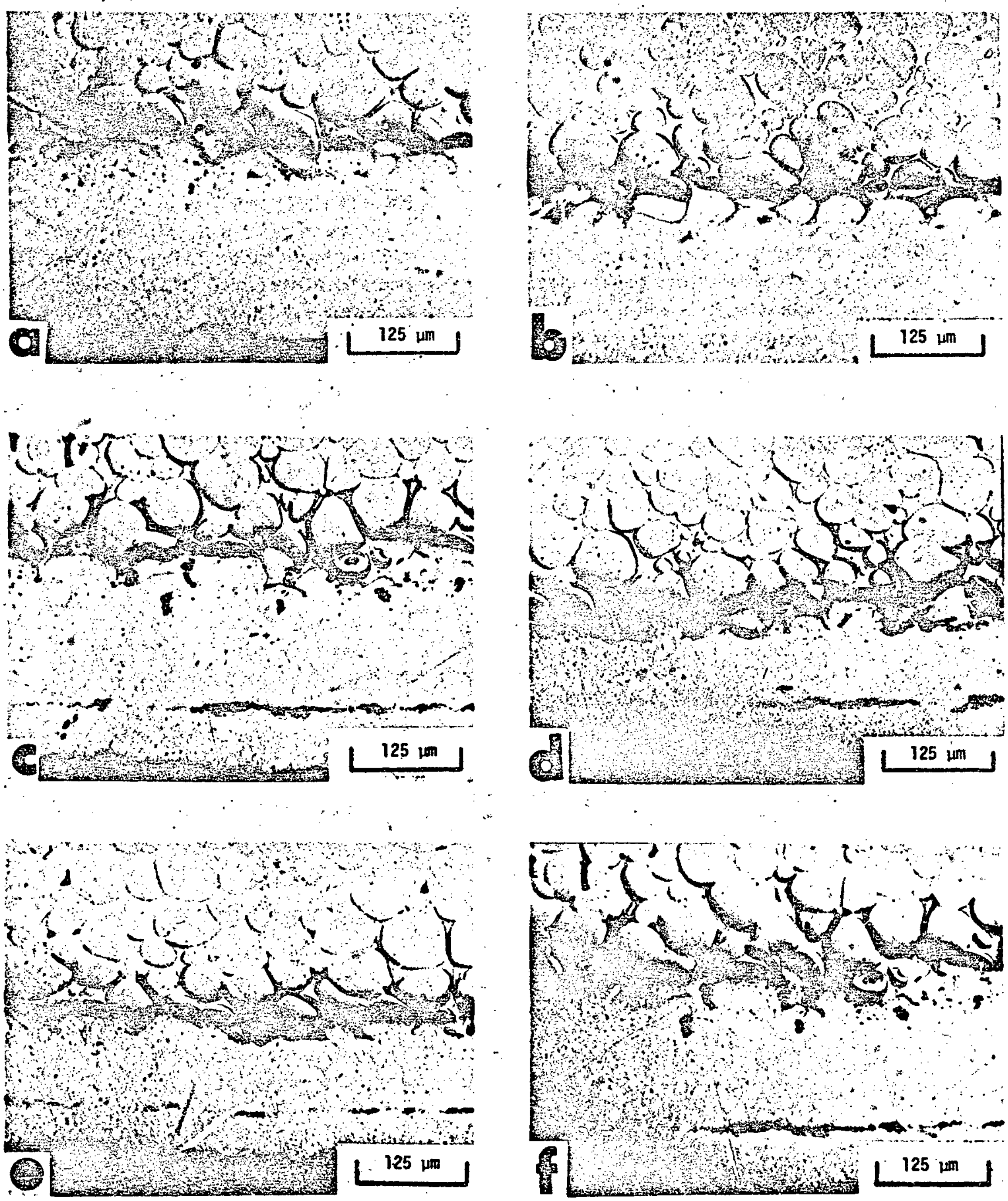


Fig. 1. Representative microstructures of the composite--copper junction showing bonding.

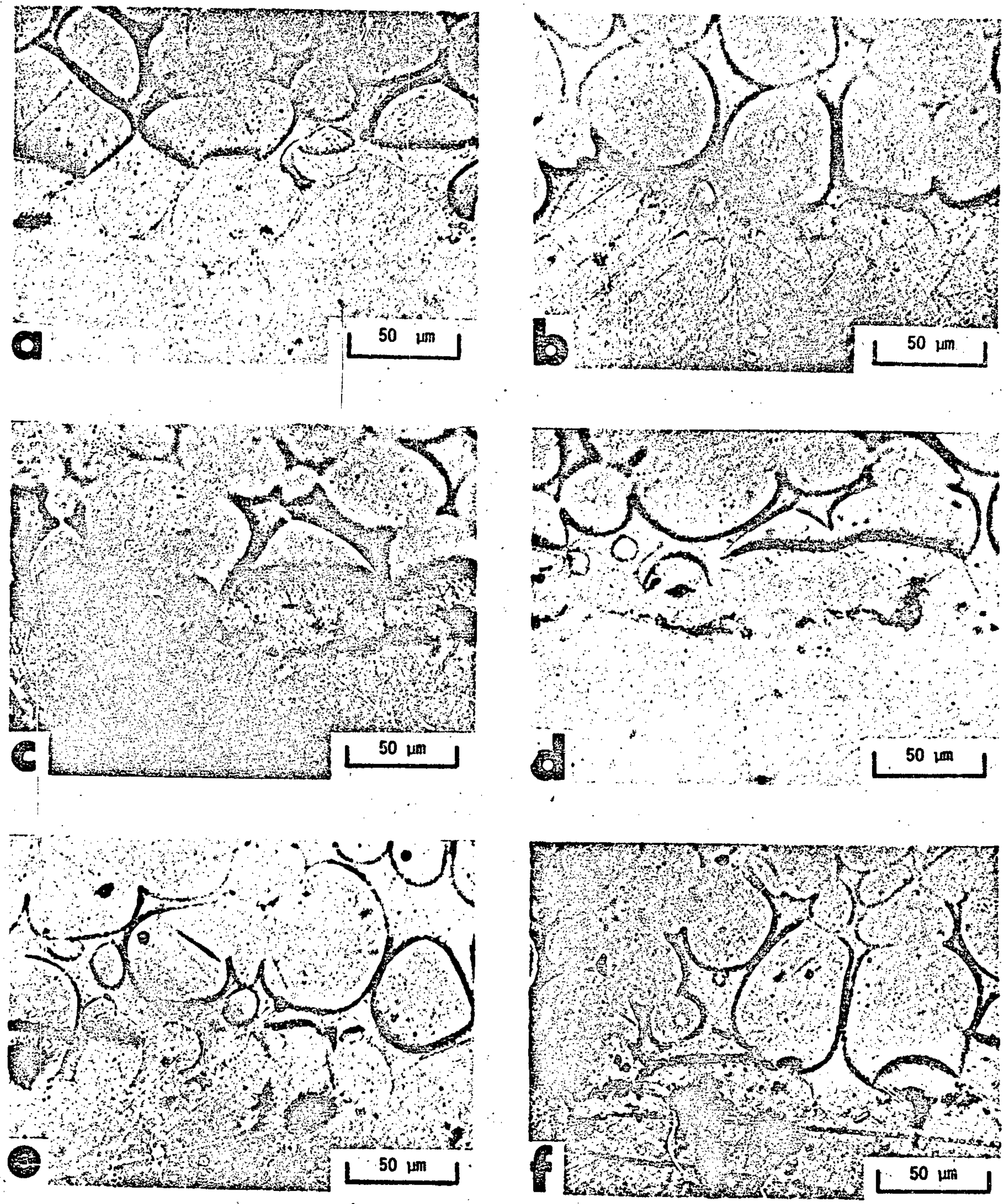


Fig. 2. Additional microstructures of the composite--copper junction.

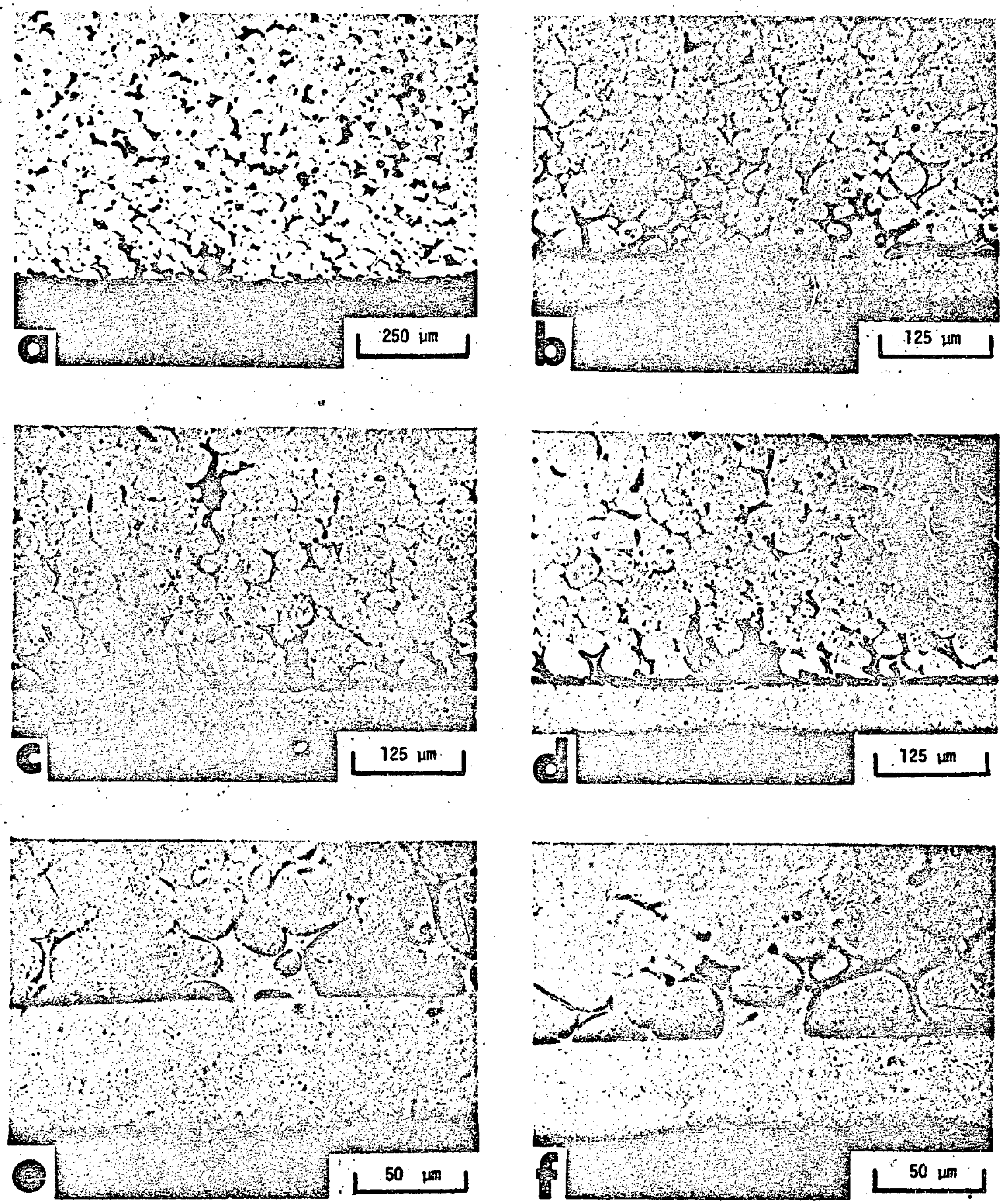


Fig. 3. Representative microstructures of the composite--nickel junction showing bonding as well as spanning of pores by the coat.

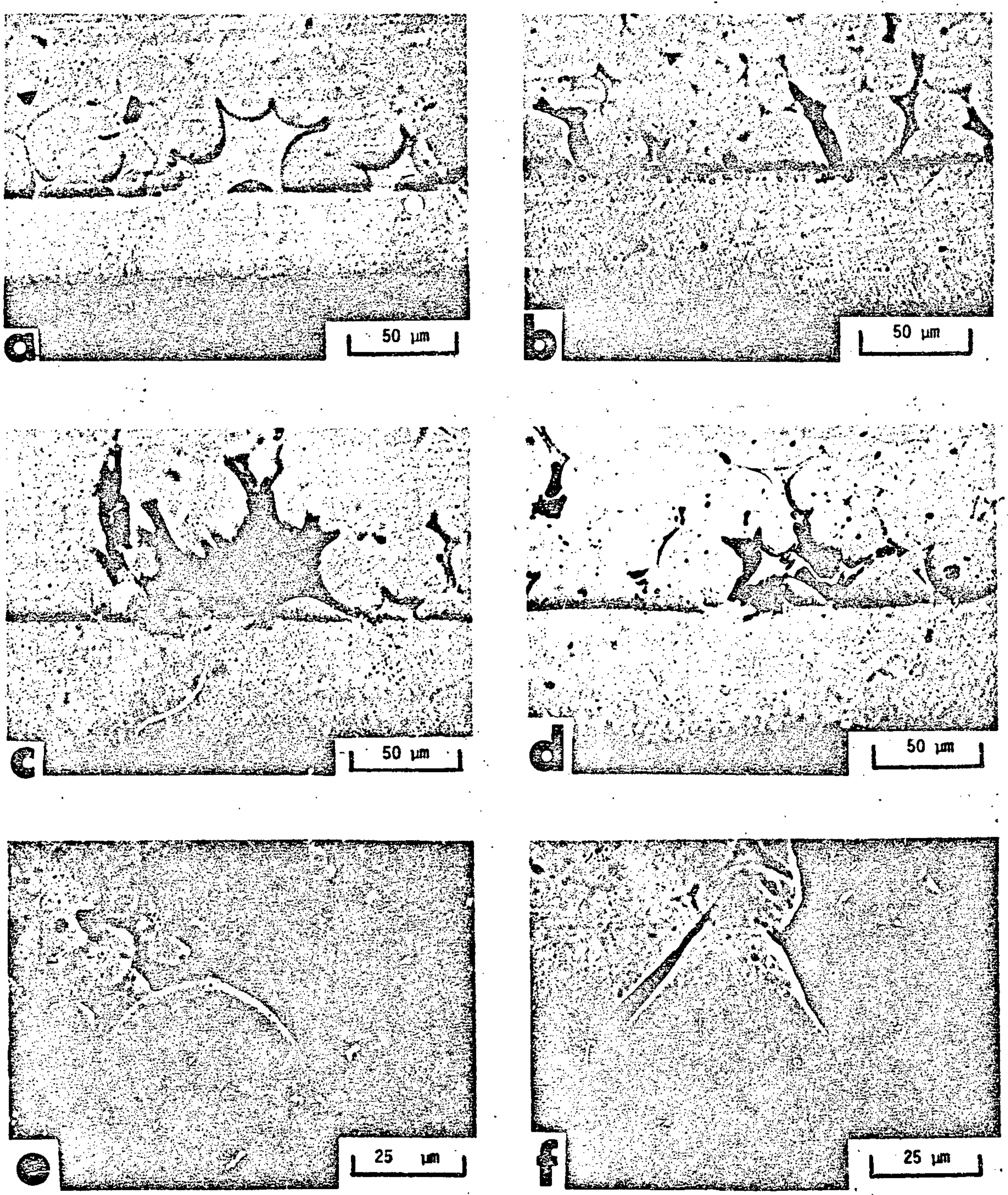


Fig. 4. Additional microstructures of the composite--nickel junction.

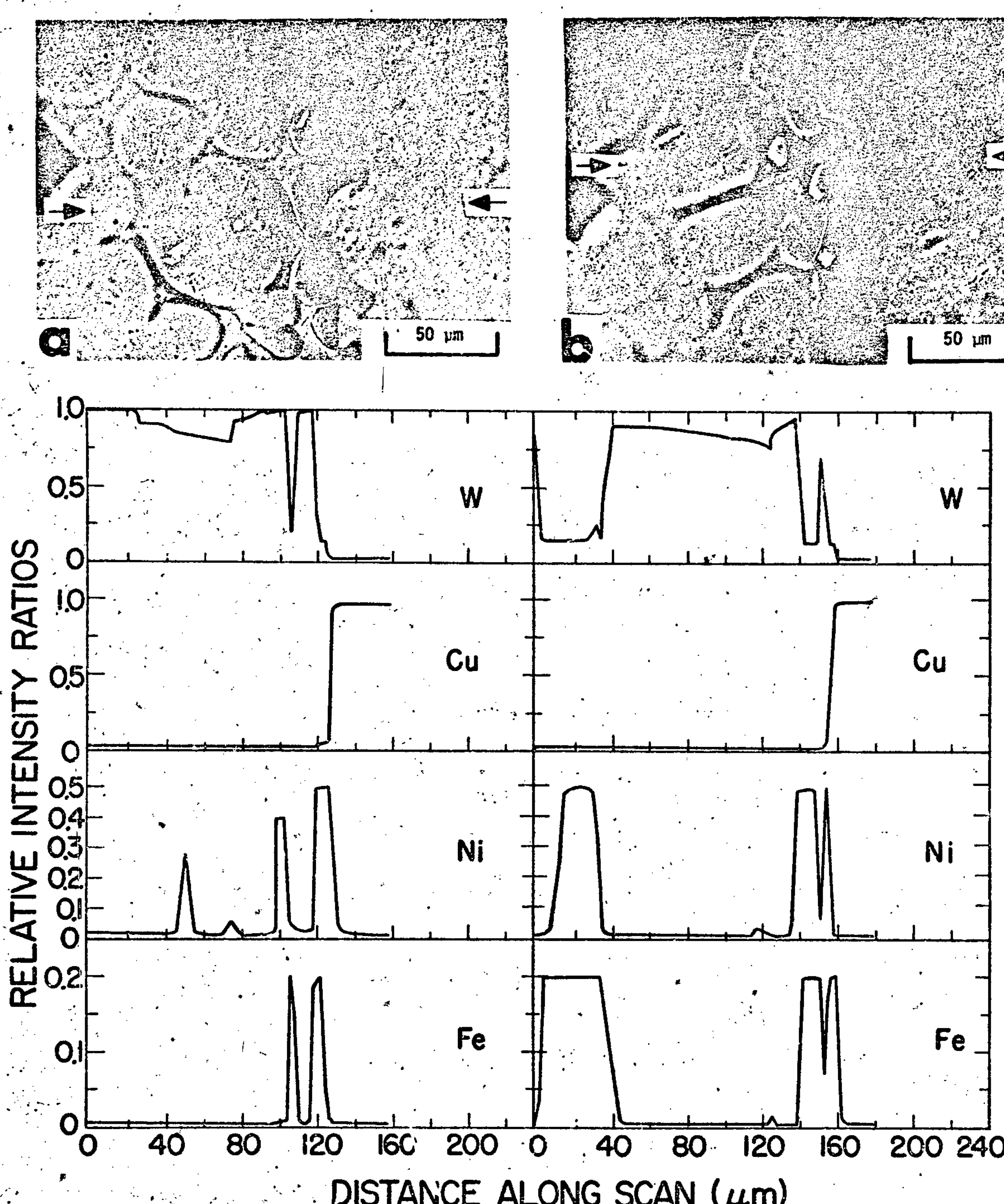
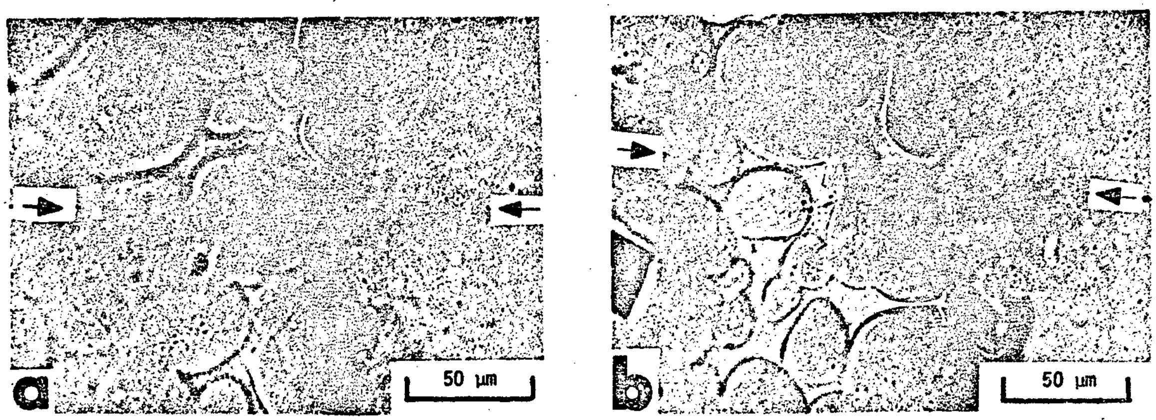


Fig. 5. Microprobe analysis of scans #1 and #2 across composite—copper junction.



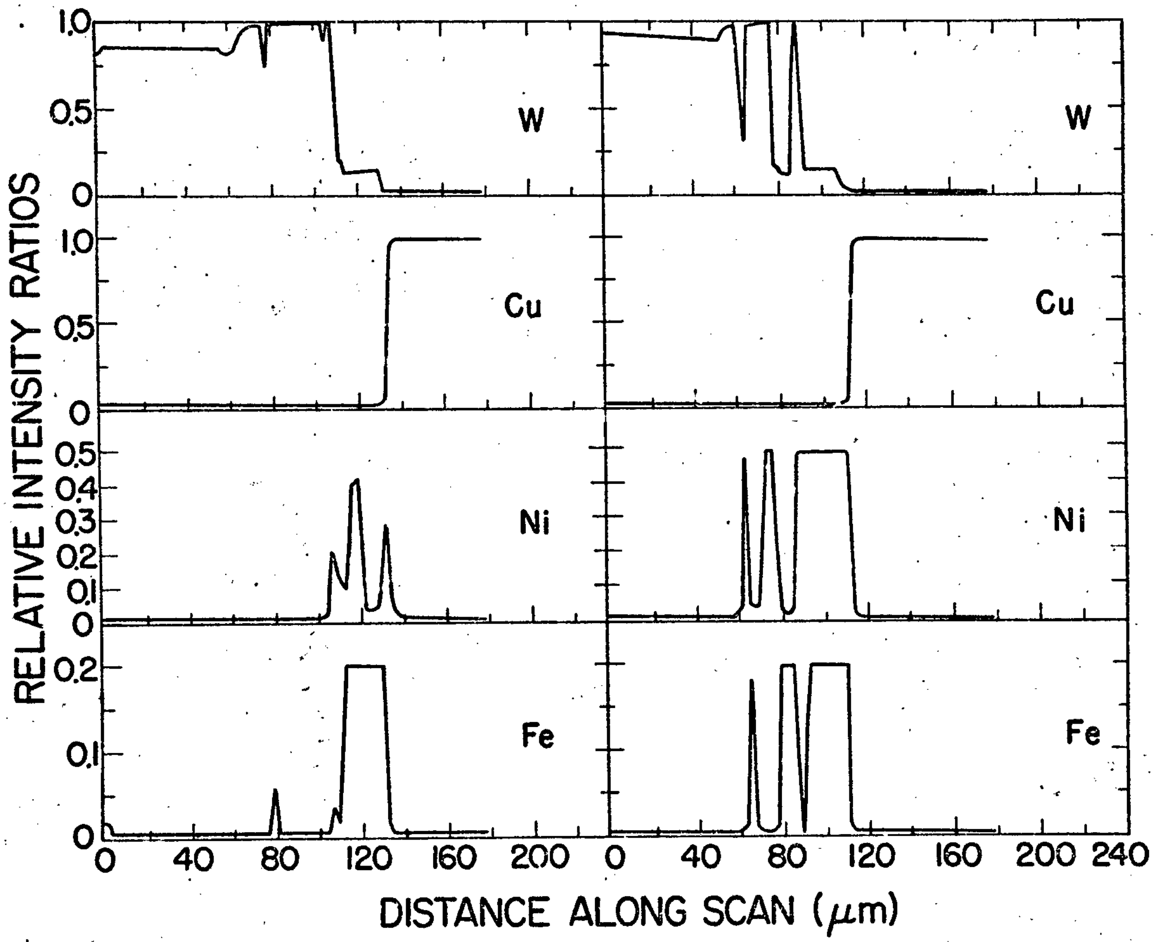


Fig. 6. Microprobe analysis of scans #3 and #4 across composite-- copper junction.

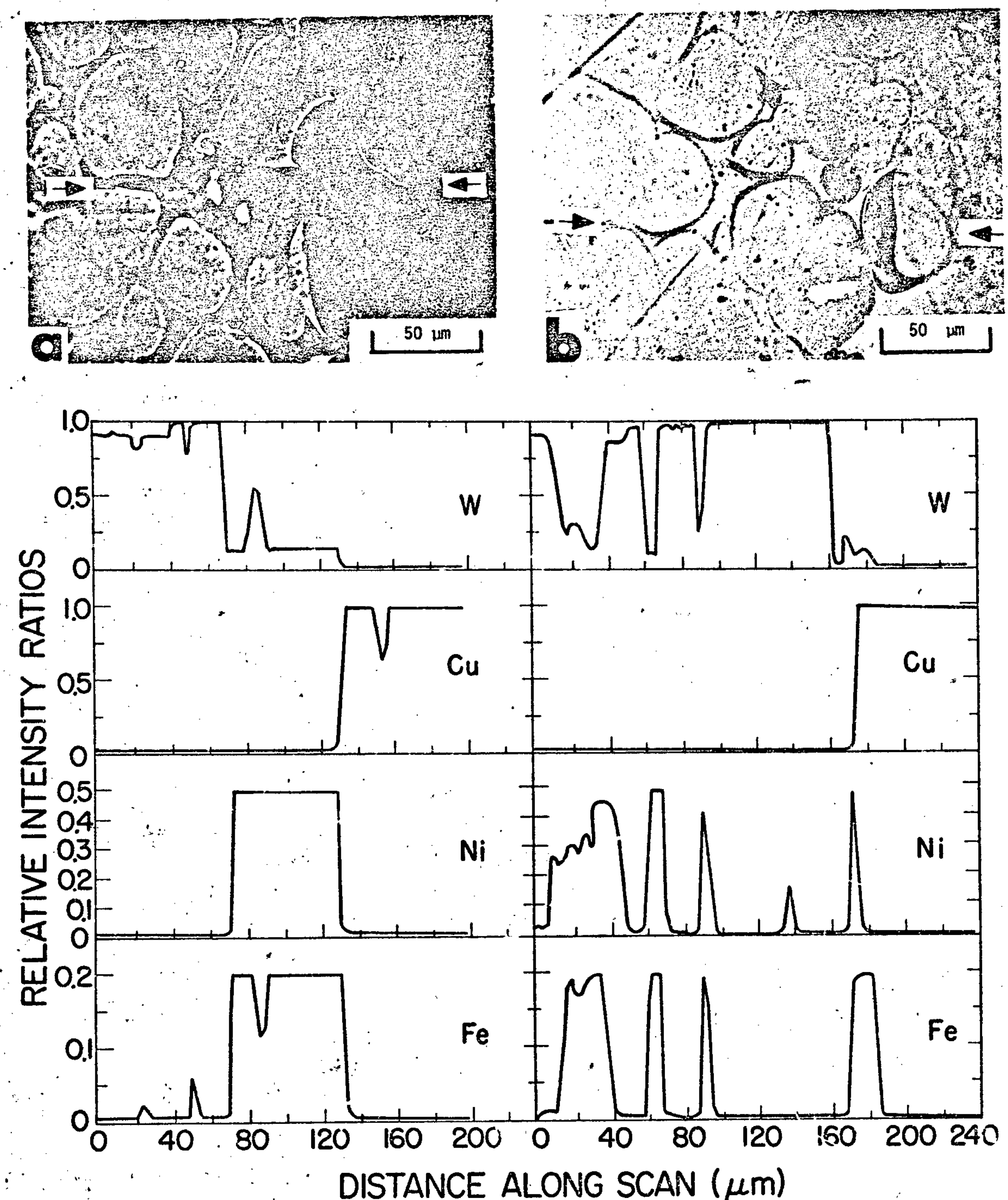


Fig. 7. Microprobe analysis of scans #5 and #6 across composite--copper junction.

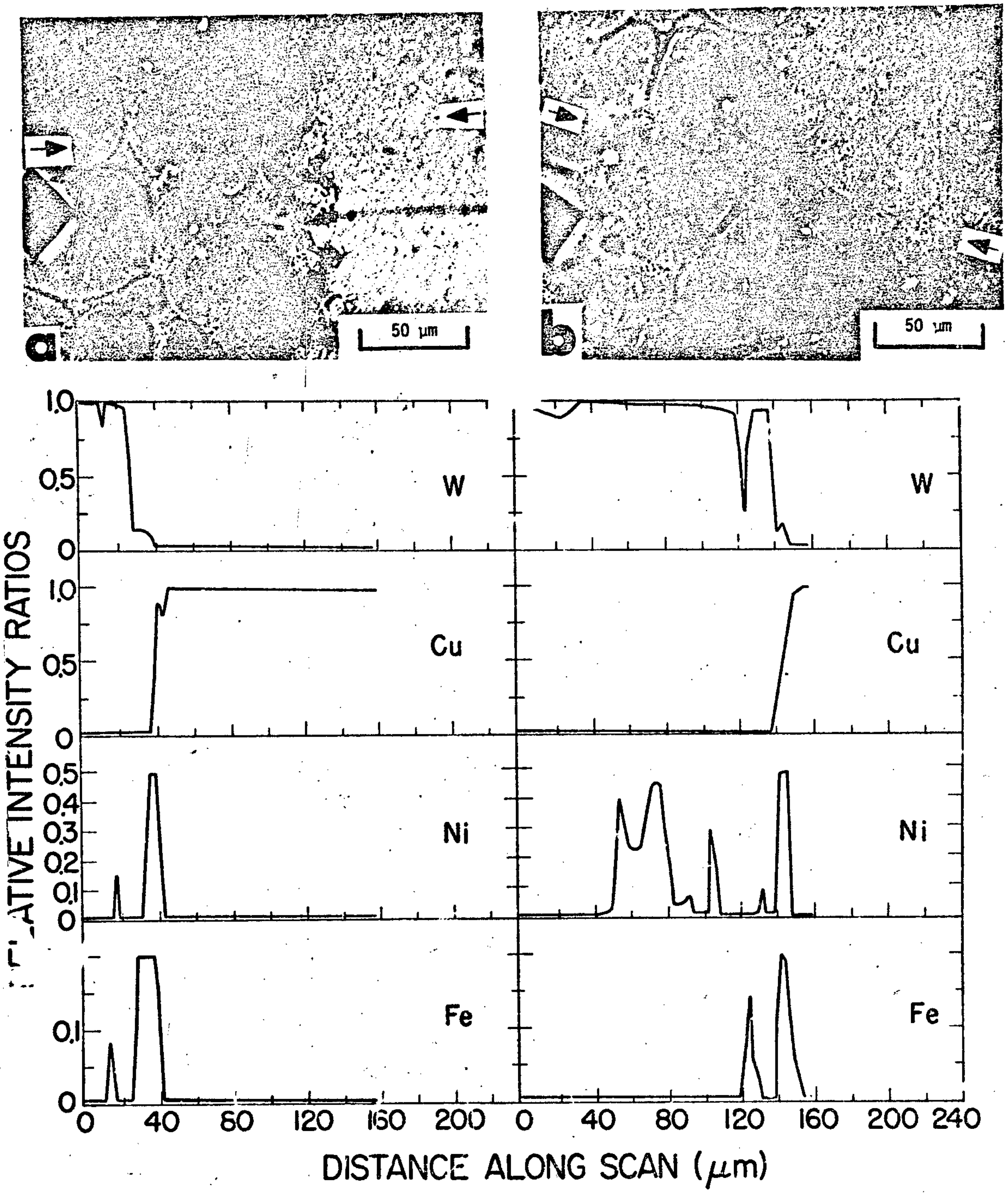


Fig. 8. Microprobe analysis of scans #7 and #8 across composite-copper junction.

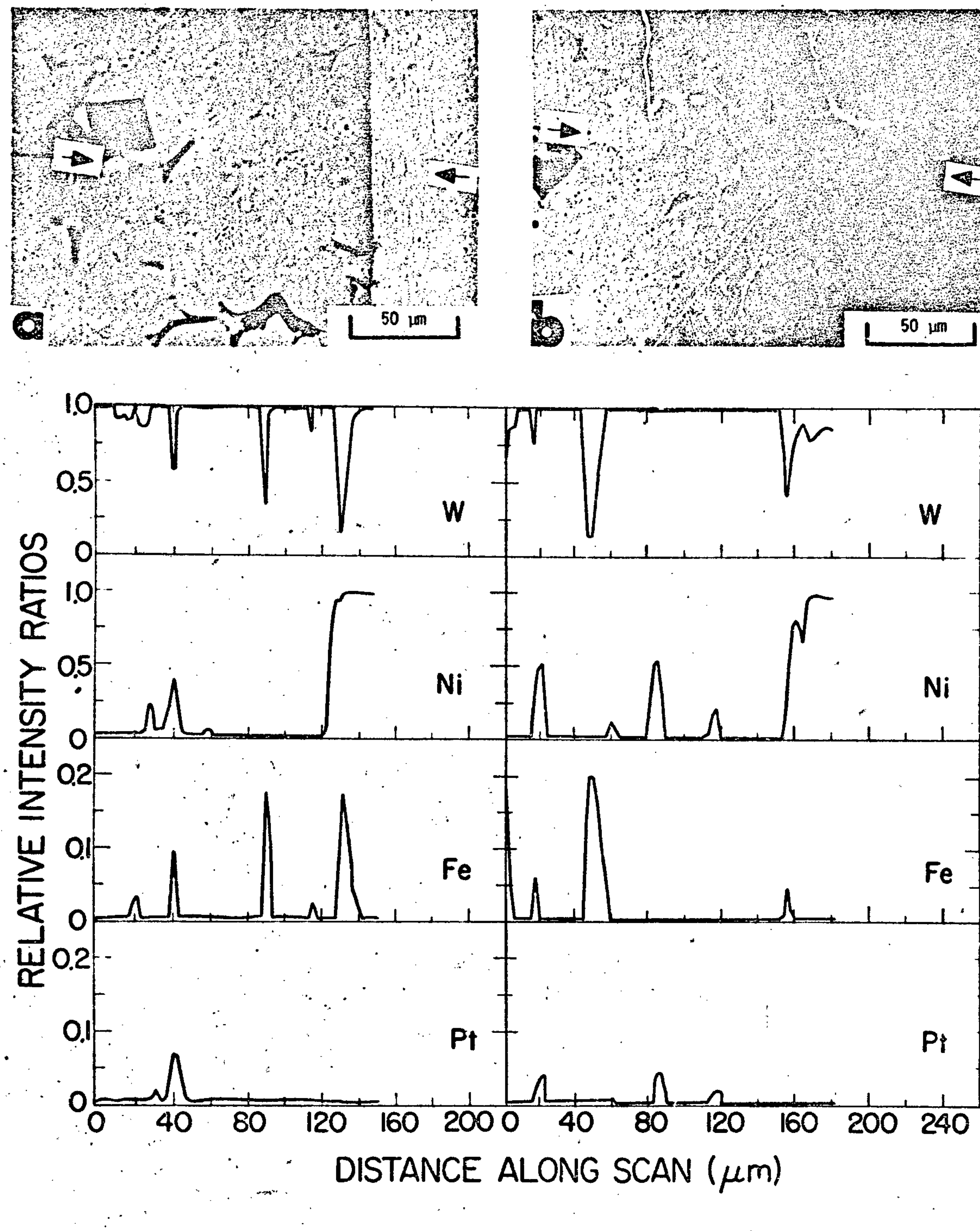


Fig. 9. Microprobe analysis of scans #1 and #2 across composite--nickel junction.

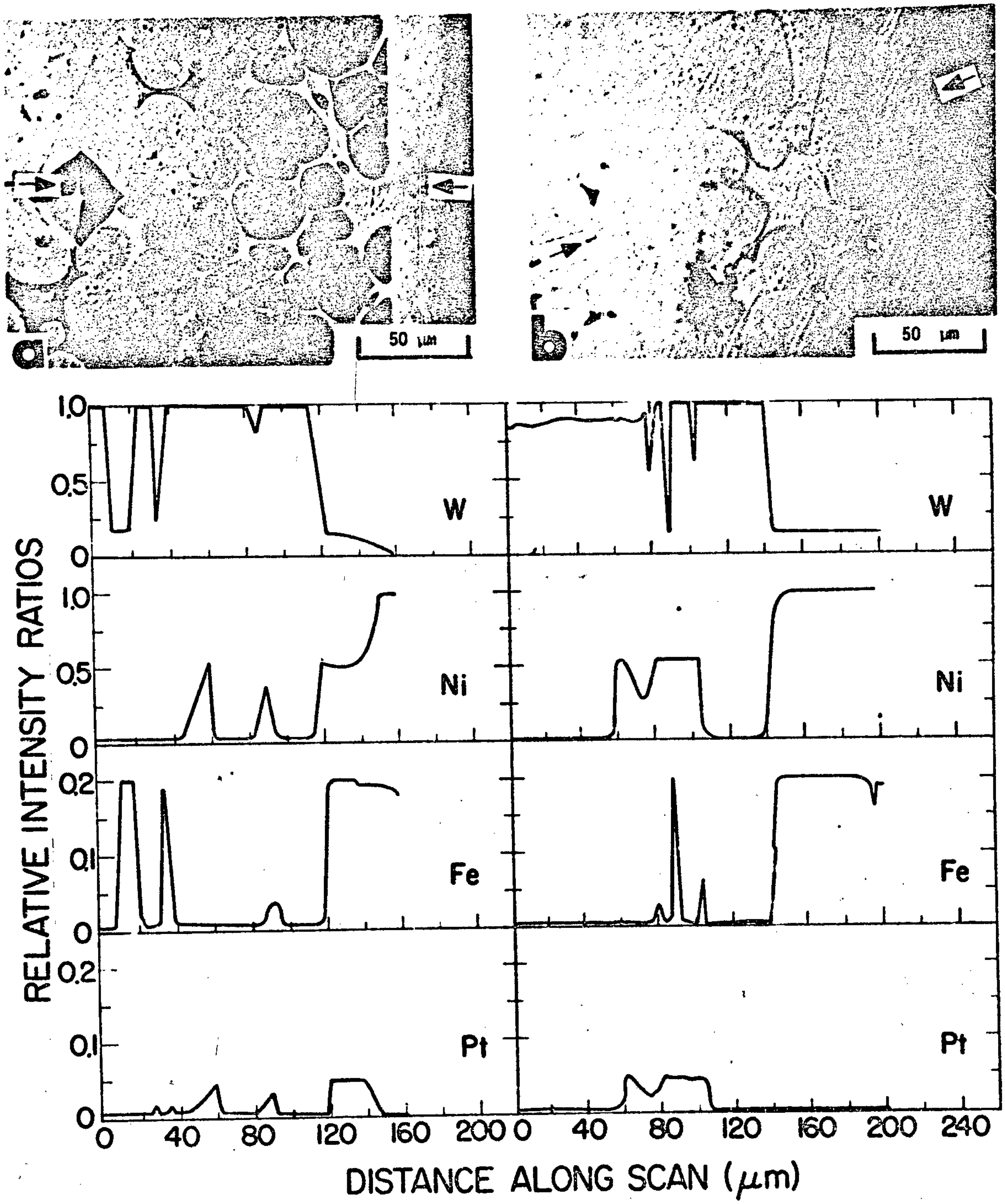


Fig. 10. Microprobe analysis of scans #3 and #4 across composite--nickel junction.

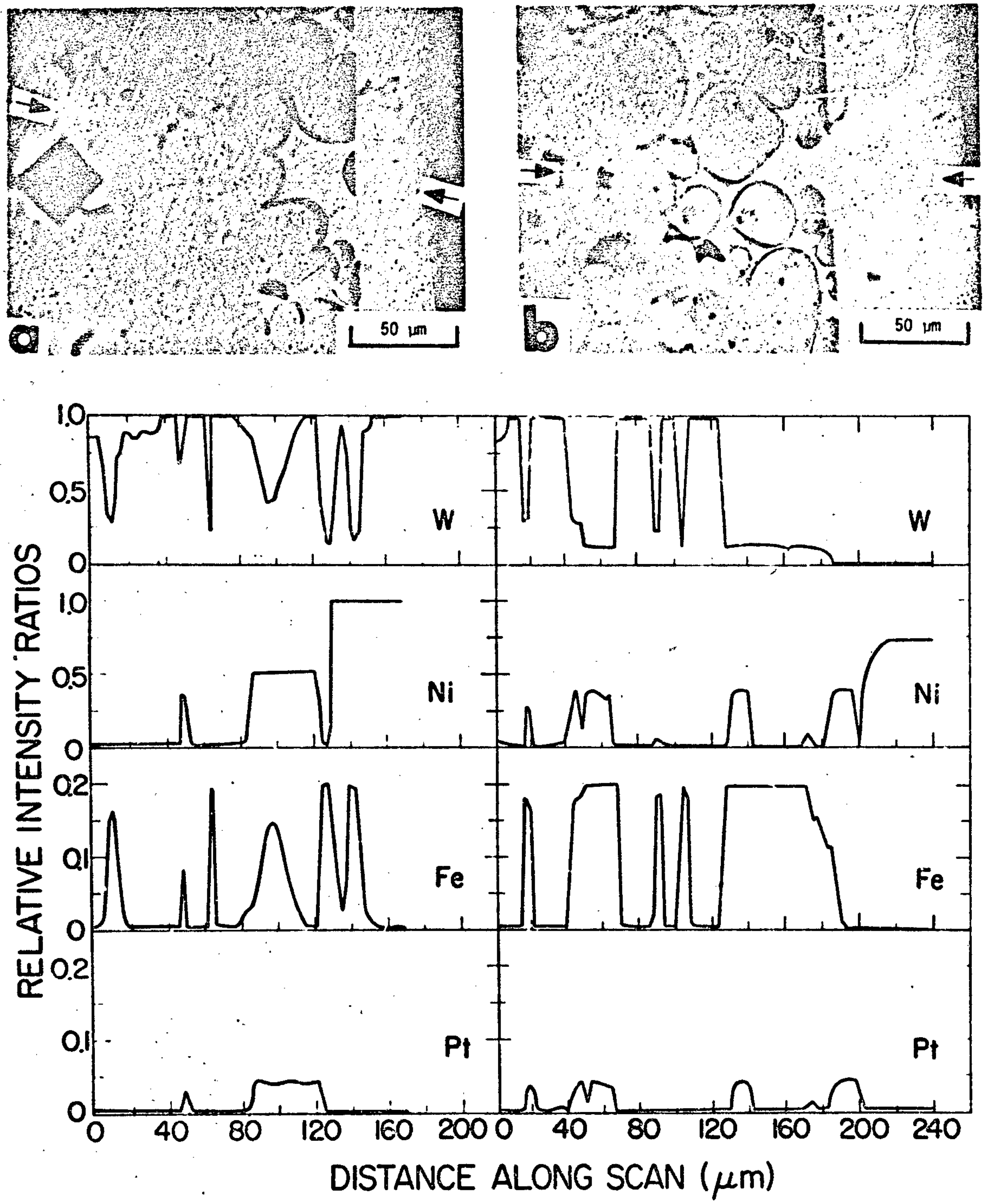


Fig. 11. Microprobe analysis of scans #5 and #6 across composite--nickel junction.

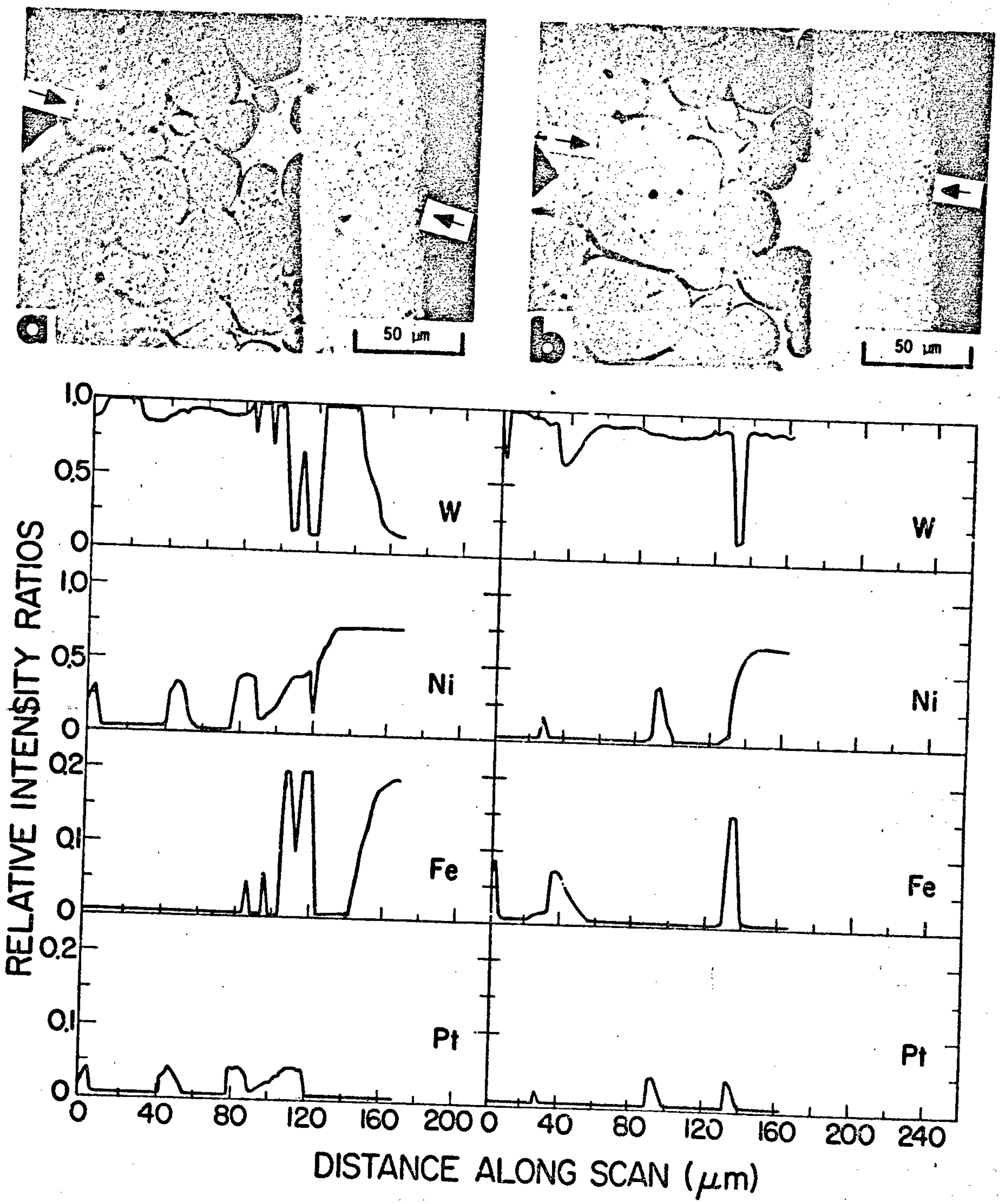


Fig. 12. Microporobe analysis of scans #7 and #8 across composite--nickel junction.