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Letter

Surface alloying of molybdenum single crystal

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Abstract

Surface alloying with rhenium was performed on a molybdenum single-crystal sheet using an electron beam of high power density.

A number of large pores formed along the weld bond. However, no definite grain boundaries were recognized by either optical microscopy or X-ray back-Laue photography. The residual content of rhenium in the fused zone was about 38 wt.% and consequently hardening occurred.

1. Introduction

In previous studies [1, 2] the microstructure and tensile properties of electron-beam- or tungsten–inert-gas- (TIG-) welded joints of molybdenum single-crystal sheet which had been prepared by means of secondary recrystallization [3] were investigated. Firstly, indications were found of a number of large pores formed in the weld metal. However, no grain boundaries were recognized in the weld metal and its crystallographic orientation was the same as in the base metal. Secondly, at temperatures below 223 K fracture initiated either at the pores in the weld metal or at the island grains in the base metal. Consequently, the strength and ductility of the welded joint were apparently comparable with those of the base metal.

In this investigation an electron beam of high power density was used to irradiate a rhenium wire which had been placed in a surface slit on a molybdenum single-crystal sheet. The microstructure and crystallographic orientation of the fused zone were examined by optical microscopy and X-ray back-Laue photography. The chemical composition and hardness in the fused zone were also measured. It is well known that rhenium addition effectively improves the low temperature ductility of molybdenum [4–8].

2. Experimental details

The material used in this investigation was a single-crystal sheet of molybdenum (about 6 mm thickness) which had been prepared by means

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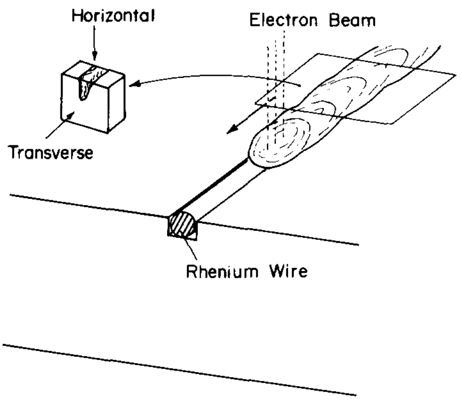

Fig. 1. Schematic diagram of surface alloying and specimen preparation.

of secondary recrystallization. This material typically contained 0.0006 wt.% C, 0.0006 wt.% O and less than 0.0003 wt.% N.

The processes of surface alloying and specimen preparation are schematically represented in Fig. 1. First a slit of 1 mm width and 0.6 mm depth was machined on the crystal surface and a rhenium wire of 1 mm diameter was placed in the slit. Then an electron beam with an acceleration voltage of 40 kV and a beam current of 200 mA was used to locally irradiate the rhenium wire, employing a traverse speed along the wire of 16.7 mm s^{-1}. Under this condition the penetration depth was about 50% of the sheet thickness. After electron beam irradiation a specimen of dimensions 4 mm × 6 mm was cut from the sheet.

Horizontal and transverse cross-sections of the fused zone were examined by optical microscopy. X-ray back-Laue photography was applied to confirm the absence of definite grain boundaries. The chemical composition in the fused zone was determined using an EDAX analyser attached to a JEOL JSM-6400. Its hardness was measured using a Vickers microhardness tester.

3. Results and discussion

The microphotograph of the transverse cross-section of the fused zone in the single crystal is shown in Fig. 2. Large pores (marked A) were observed along the weld bond that form the interface between the fused zone and the base metal. In addition, "boundary-like" traces (marked B) were observed in the vicinity of the centre of the fused zone. For comparison, in polycrystalline molybdenum without rhenium a coarse cast structure had developed and a macrocrack had been generated just along the centre of the fused zone. In order to examine the boundary-like trace in more detail, X-ray back-Laue photography was applied. The fused zone of a single crystal ("×" indicates the analysed position) demonstrated exactly the same group of Laue spots as the base metal, though each spot for the fused zone was slightly distorted.

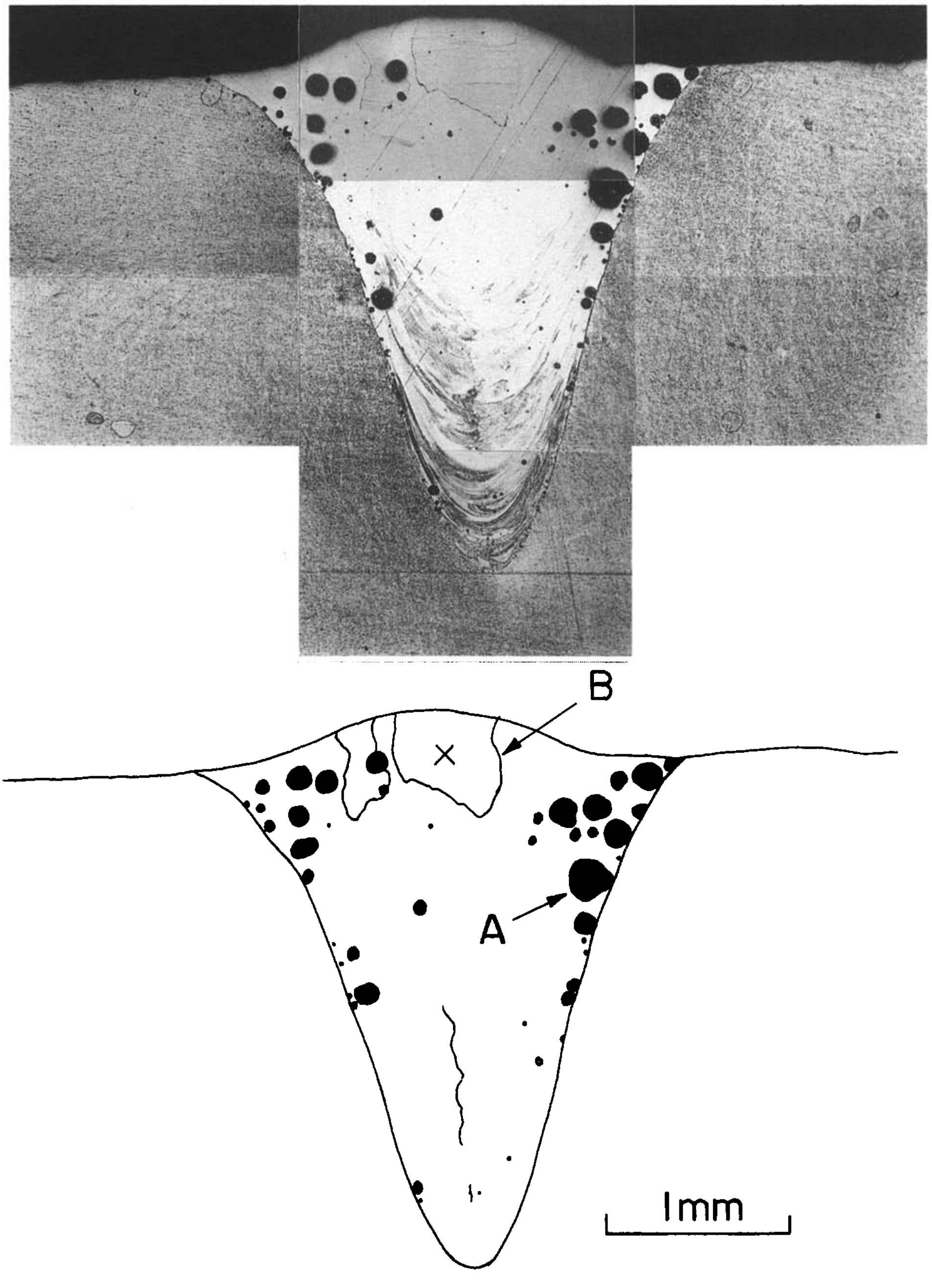

Fig. 2. Microphotograph of the fused zone in single-crystal molybdenum with rhenium wire.

Therefore it might be concluded that the boundary-like trace is not a definite grain boundary but a small misfit angle boundary or a subgrain. The distortion of the Laue spots is due to the lattice distortion resulting from the rhenium

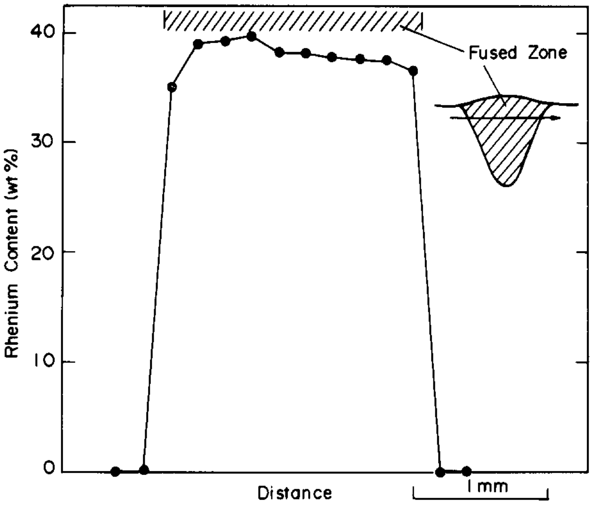

Fig. 3. Rhenium content in the fused zone of single-crystal molybdenum with rhenium wire.

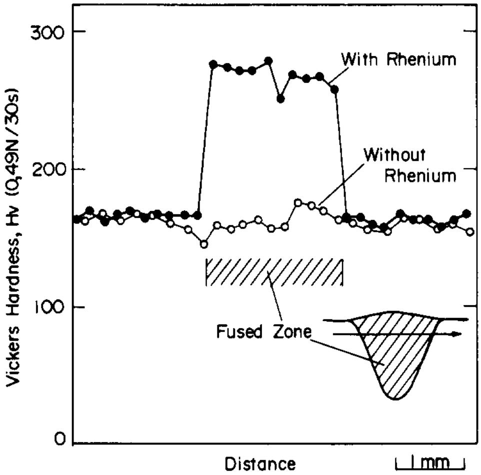

Fig. 4. Vickers hardness in the fused zone of single-crystal molybdenum with and without rhenium wire.

alloying with different crystal structure and lattice constant, in addition to the residual stress resulting from the large heat input.

The rhenium content in the fused zone is shown in Fig. 3. In the present investigation the rhenium content (C_{Re}) was determined by using the EDAX analyser without the standard sample. It is shown that the rhenium distribution is relatively uniform and the average value is about 38 wt.%.

Assuming that 100% Re is preserved on electron beam irradiation, the residual rhenium content (C^*_{Re}) in the fused zone is roughly expressed by the equation

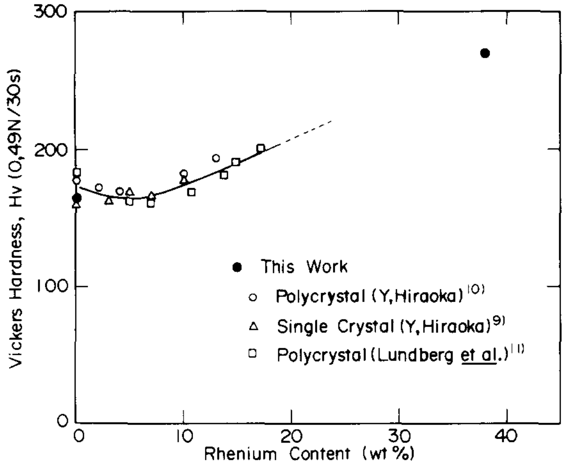

Fig. 5. Plot of hardness as a function of rhenium content.

$$C^*_{\mathrm{Re}} = \frac{A_{\mathrm{Re\ wire}}}{A_{\mathrm{fused\ zone}}} \times 100$$

where $A_{\mathrm{Re\ wire}}$ and $A_{\mathrm{fused\ zone}}$ are the cross-sectional areas of the rhenium wire and the fused zone respectively. By introducing the experimental values for $A_{\mathrm{Re\ wire}}$ and $A_{\mathrm{fused\ zone}}$, C^*_{Re} is calculated to be 35 wt.%. Therefore it is deduced that the yield of rhenium during electron beam irradiation is approximately 100%.

The hardness distribution in the fused zone is shown in Fig. 4. It is obvious that rhenium addition has induced hardening. In Fig. 5 the hardness is plotted against the rhenium content. For reference, the data obtained for a single crystal [9] and a polycrystal [10, 11] of Mo–Re alloy are also plotted in this figure.

4. Summary

By using an electron beam of high power density, surface alloying of single-crystal molybdenum was performed.

A number of large pores formed in the fused zone but no definite grain boundaries were recognized. Rhenium addition of 38 wt.% was achieved and hardening occurred.

Acknowledgments

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References

1 Y. Hiraoka, T. Fujii, M. Okada and R. Watanabe, *J. Jpn. Inst. Met.*, *9* (1984) 945.

2 Y. Hiraoka and T. Fujii, in H. Bildstein and H. M. Ortner (eds.), *Proc. 12th Int. Plansee Seminar '89, Reutte/Tirol, 1989*, Metallwerk Plansee GmbH, Tirol, 1989, p. 265.

3 T. Fujii, R. Watanabe, Y. Hiraoka and M. Okada, *J. Less-Common Met.*, *96* (1984) 297.

4 G. A. Geach and J. R. Hughes, in F. Benesovsky (ed.), *Plansee Proc.*, Pergamon, Oxford, 1956, p. 245.

5 R. I. Jaffee, C. T. Sims and J. J. Harwood, in F. Benesovsky (ed.), *Plansee Proc.*, Pergamon, Oxford, 1959, p. 380.

6 A. Lawley and R. Maddin, *Trans. Am. Inst. Miner. Eng.*, *224* (1962) 573.

7 E. M. Savitskii, M. A. Tylkina and K. B. Povarova, *Rhenium Alloys*, Nauka, Moscow, 1965 (*Transl. JPRS No. 34*, U.S. Department Commerce, March 1966, p. 566).

8 D. L. Davidsonn and F. R. Brontzen, *J. Appl. Phys.*, *39* (1968) 5768.

9 Y. Hiraoka, submitted to *J. Jpn. Inst. Met.*

10 Y. Hiraoka, unpublished data, 1990.

11 L. B. Lundberg, E. K. Ohriner, S. M. Tuominen, E. P. Whelan and J. A. Shields Jr., in K. H. Miska, M. Semchyshen and E. P. Whelan (eds.), *Physical Metallurgy and Technology of Molybdenum and Its Alloys*, AMAX, Ann Arbor, MI, 1985, p. 71.