Liquid Emulsion Autoradiography of Metals

by Jack C. Bokros and Philip C. Rosenthal

DEVELOPMENT of the autoradiographic method in metallurgical research has been directed mainly toward gaining higher resolution, i.e., toward being able to locate segregation of a radioactive isotope on a finer scale. In the investigation reported, the liquid emulsion autoradiographic technique used in the biological science was adapted to the autoradiography of metals. The distribution of isotopes in metals was studied using this technique.

Amorphous carbon uniformly labelled with carbon-14 at a specific activity of 6.95 microcuries per millimole was obtained from Tracerlab Inc. Carbon-14 emits β particles having a maximum energy of 157 kev and an average energy of about 50 kev. These βs have a maximum range of about 30 mg per cm² and a half-thickness of about 3 mg per cm².

The iron used was spectroscopically pure electrolytic iron prepared by the Oak Ridge National Laboratory.

Unhardened nuclear track emulsion, type NTB², was kindly supplied by the Eastman Kodak Co. This emulsion contains approximately 80 pct Ag halide, carbon-14 in pure iron after various treatments was studied using this technique.

Reference

ethyl ketone, which gave a plastic film on the metallographic specimen about 0.01" thick. The specimen was removed all of the solvent.

Specimens were dipped in a solution of two parts vinyl acetate dissolved in 100 ml of methyl acetate, heated at 100°C for about 30 min in an oven to melt the track emulsion. The specimens were then placed in a light-tight container along with a small beaker of CaSO₄. The processing solutions were kept at 18°C.

After a 10 day exposure period, the specimens were removed and developed. Because these emulsions disintegrate rapidly in aqueous solutions above 20°C, the processing solutions were kept at 18°C. The autoradiographs were developed for 4 min in Eastman Kodak D-19 developer and fixed in Eastman Kodak F-5 acid fixer with hardener for twice the time to clear. The autoradiographs were dried in a gentle air current. At this point the emulsions wrinkled if they were too thick, resulting in the displacement of the image from the active areas by a small amount; however, when the emulsions were thin enough to clear in about 30 sec in the F-5 fixer, no wrinkling or shifting occurred.

An illustrative autoradiograph shown in Fig. 1 is of pearlite at X100. Fig. 2 is an autoradiograph of the martensite-ferrite interface obtained by carburizing below the transformation temperature. The autoradiograph shown in Fig. 3 shows very high grain boundary activity produced in ferrite by annealing a carburized specimen for 138 hr at 538°C followed by a water quench. This iron specimen had been carburized at 838°C for 91 hr and water quenched before the 138 hr anneal. The grain boundary activity varied from boundary to boundary, indicating a possible orientation effect. This grain boundary activity is probably due to the solubility of carbon in ferrite at annealing temperature.
 Autoradiographs of martensite indicate a random distribution of carbon in austenite with no abnormal grain boundary effects. None of the autoradiographs of specimens carburized above the α→γ transformation temperature in pure iron gave any evidence of accelerated grain boundary diffusion of carbon in austenite.

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References

Technical Note

Effect of Cooling Rate on Hardness of Commercial Titanium Alloys
by Howard Martens

HARDNESS behavior of commercial titanium alloys following various heat treating processes has been studied for some time. However, the hardness of such alloys following a definite measured cooling rate from the single phase β region has not been reported. Therefore, a series of experiments was conducted on the three alloys: 150A, 130A, and 130B. The manufacturers gave the following compositions for these alloys: Ti 150A: 0.045 pct C, 0.069 pct N, 1.32 pct Fe, and 2.68 pct Cr; RC 130A: 0.13 pct C, and 7.9 pct Mn; RC 130B: 0.11 pct C, 3.5 pct Mn, and 3.2 pct Al. A method similar to that described by Greninger was used, and the cooling rate varied from 6" to 9200°F per sec. The cooling curves were recorded on a Speedomax or a recording oscillograph, and the hardness was measured on a Tukon hardness tester using a Knoop indenter with a 500 g load.

The curves of Fig. 1 show the variation of the Knoop hardness with the cooling rate for these three alloys. The alloys 150A and 130B behave in a similar manner, as shown by the curves. In neither of these alloys was it possible to retain the high temperature β phase, even by the most rapid cooling rate. The slow cooling rates produced a typical α+β structure in these two alloys. As the cooling was increased, these phases became more finely dispersed and the hardness increased. This increase in hardness with cooling rate continued for the 150A alloy until a rate of approximately 1000°F per sec was reached. At this cooling rate, the structure showed the result of the decomposition of the β phase into the supersaturated α solid solution which is referred to as α'. At higher cooling rates, the structure showed no change and the hardness showed no great change, as indicated by the dotted portion of the curve in Fig. 1. The increase in hardness with cooling rate for the 130B alloy continued until a rate of approximately 300°F per sec was reached. At this rate the α' structure was produced and the maximum hardness was reached. Higher cooling rates caused no marked changes in hardness or structure.

The alloy 130A behaved in a different manner, as shown by the curve of Fig. 1. The structure produced by the slow cooling rates was a typical α+β structure, which became finely dispersed as the cooling rate increased. The hardness increased with

Fig. 1—Curves show the variation of Knoop hardness with cooling rates from 2000°F for alloys Ti 150A, RC 130A, and RC 130B.

cooling rate and reached a maximum at approximately 120°F per sec. At this maximum hardness, the structure showed the result of the decomposition of the β phase into the β+α' phase structure. As the cooling rate was increased, smaller amounts of the α' phase were formed and the alloy became softer. For cooling rates of 3000°F per sec and above, the high temperature β phase was retained and the hardness of the alloy did not change markedly.

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