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Creep Fracture Phenomena in Iron Embrittled by Liquid Copper

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Tensile creep studies of the embrittlement of notched iron by liquid copper in the range 1100 to 1130°C have shown the embrittlement to be of the delayed failure type, from diffusion-controlled, grain boundary penetration by copper with accompanying extended surface notching. Linking of surface cracks and internal cavities along copper-penetrated grain boundaries was the final failure mode. Crack growth was determined by the rate and amount of copper diffusion ahead of a crack and not by the feed of liquid to the crack tip.

1. Introduction

Liquid metal embrittlement of otherwise ductile metals has been extensively reviewed [1, 2]. There is considerable evidence to show that the commonest phenomena associated with embrittlement include: liquid metal atom adsorption-induced cracking; delayed failure from grain boundary penetration of solid by liquid; and the stress-aided dissolution of metal from a crack tip. The particular role of the liquid phase, therefore, may be as an adsorbent, a penetrant or a solvent operating at a crack tip.

There is little doubt that in embrittlement the liquid metal adsorbs alongside strained atomic bonds, thereby lowering the binding energy of the atomic bonds which define the crack tip [3]. The surface energy associated with the nucleation and growth of a crack is therefore lowered [4]. However, if plastic flow of the metal accompanies crack growth then the fracture stress may not be greatly altered by the reduced surface energy. The principal effect of simultaneous crack growth and metal flow is to alter the shape of the crack tip [3].

In the present work with polycrystalline iron (notched at the surface to contain liquid copper and to initiate cracking), the liquid copper was essentially a penetrant. The main objective of our work was to establish the mechanism by which copper influenced crack growth from the notch during the simultaneous processes of creep deformation and copper diffusion.

2. Experimental Procedure

Constant load, creep test specimens of pure iron (AO grade supplied by BISRA), 25 mm gauge length, 5 mm diameter with screwed ends were machined to form a central, circumferential notch (either 1 mm deep, 1 mm wide or 0.5 mm deep, 0.5 mm wide) which acted as a convenient reservoir for liquid copper. A thin copper coating (8.5 μm thick) was applied to the notch surface using a cyanide bath. Greater volumes of liquid copper (up to 0.8 notch depth for the 0.5 mm deep, 0.5 mm wide notch) were obtained from wrapping pure copper wire, 0.18 mm diameter (supplied by Johnson, Matthey and Co Ltd) around the notch.

The experimental work was carried out at 1100 to 1130°C (viz. above 1094°C, the peritectic temperature of the iron–copper system) to ensure that the copper-rich embrittling liquid remained molten during the creep test. Stress-rupture tests were then performed to record time to failure and specimen elongation for different applied stresses (0.6 to 0.8 tons/in.^{2}) and different notch volumes of liquid copper. In each test, the load was applied immediately the test temperature was attained to minimise intergranular diffusion by liquid copper before creep testing.

After failure, each specimen was cooled quickly to ~ 500°C and more slowly cooled to room temperature in an inert atmospere of dried, purified argon before sectioning for metallographic examination.

Creep specimens were sectioned longitudinally on a spark machine and hot mounted in bakelite powder. The surface was metallographically prepared by wet grinding on 0, 00, 3/0 and 4/0 grades of silicon carbide papers with final polishing on automatic polishing wheels using "metron A" cloths impregnated with diamond compounds of 6, 1 and $\frac{1}{4}$ μm grades.

Fractured surfaces of both pure iron and copper-coated iron specimens were examined by scanning electron microscopy. Degreased specimens were held firm by inserting into a 5 mm diameter hole drilled in the centre of the specimen holder. A thin conducting film of silver (500 Å) was evaporated on to the fractured surfaces. Also a conducting media of "Aquadag" (colloidal graphite in water) was pasted around the base of the specimen to form a good conducting path between the specimen and the holder.

3. Results and Discussion

Preliminary experimental results [5] showed that the overall creep rate of copper-coated iron was considerably greater than that of pure iron.

Metallographic evidence from interrupted tests indicated that during creep, liquid copper penetrated along the grain boundaries at the tip of the notch probably promoting either grain boundary sliding or rotation. Bentle and Kniefel [6] also concluded that the probable influence of MgF_2 liquid at the grain boundaries of BeO-MgO specimens was to increase grain boundary sliding. Our results also showed that the time to failure and specimen elongation was considerably less for the copper-coated iron specimens than for pure iron.

Metallographic evidence from interrupted tests in the steady-state stage confirmed the presence of surface cracks associated with copper-penetrated grain boundaries below the notch (fig. 1). The liquid copper-rich phase appeared to be ahead of the growing surface cracks. In order to confirm whether crack growth was dependent or not upon there being a feed of liquid copper to the crack tip, some iron specimens were prestrained to initiate cracks below the notch, coated with copper and then creep tested. Metallographic evidence from interrupted tests revealed the copper-rich phase ahead of the preformed cracks. Thus the rate of copper penetration was faster than the rate of surface crack growth. The depth of surface cracking was found to be controlled by the depth of copper penetration. It is probable that the copper-rich

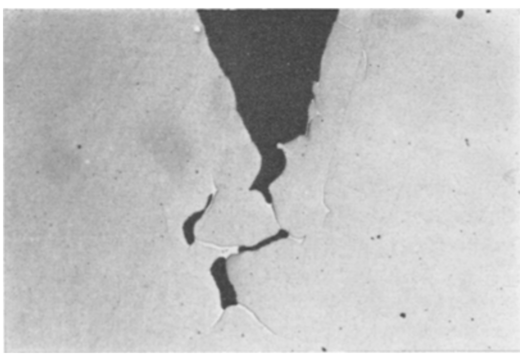

Figure 1 Iron with a 0.5 mm deep, 0.5 mm wide notch containing 0.29 notch depth of copper, creep tested at 1100°C under an applied stress of 0.55 tons/in^2 at the notch (creep strain 2.8%; creep time 156 min.; steady-state stage), showing the presence of surface cracks associated with copper-penetrated grain boundaries below the notch (× 25.2 (Enlarged × 2) Unetched).

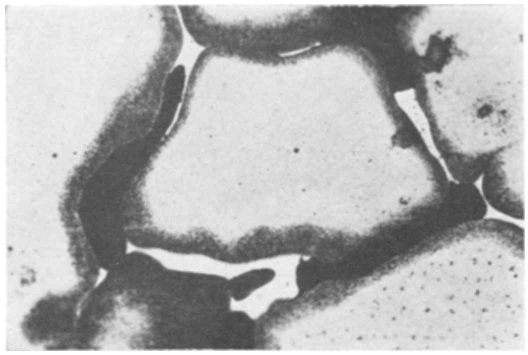

Figure 2 Iron with a 0.5 mm deep, 0.5 mm wide notch containing 0.29 notch depth of copper, creep tested at 1100°C under an applied stress of 0.55t.s.i. (creep strain 4.0%; creep time 246 min; steady-state stage), showing a grain near the notch area which was isolated by the linking up of cracks and liquid phase (× 100 (Enlarged × 2) Etched in 2% nital reagent).

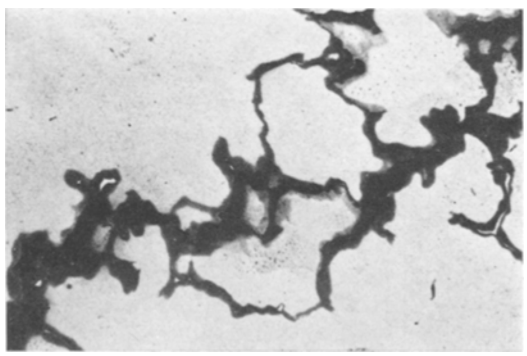

Figure 3 As fig. 2, but at a later time in creep (creep strain 5.6%; creep time 256 min; tertiary stage of creep), showing surface cracks and cavities which extended along the copper-rich phase and eventually linked up (×25.2 (Enlarged × 2) Etched in 2% nital reagent).

areas provided a low energy path for crack growth during steady-state creep.

There was additional evidence that both surface cracks and internal cavities were associated with the copper-penetrated grain boundaries.

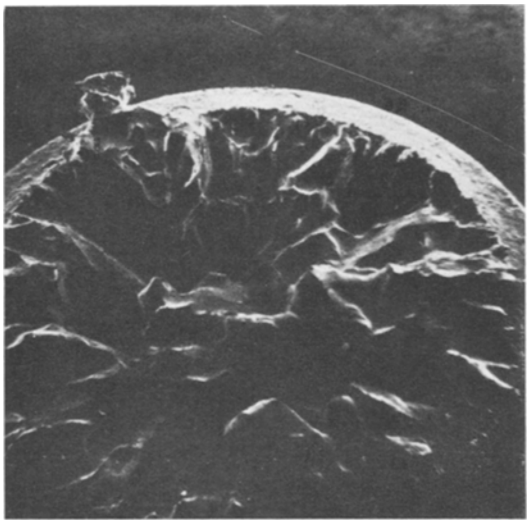

Figure 4 Scanning electron micrograph of the fracture surface of an iron specimen with a 0.5 mm deep, 0.5 mm wide notch containing 0.29 notch depth of copper, creep tested at 1100° C under an applied stress of 0.55 tons/in^2 at the notch (time to failure 370 min.; elongation 5.9%) (× 24).

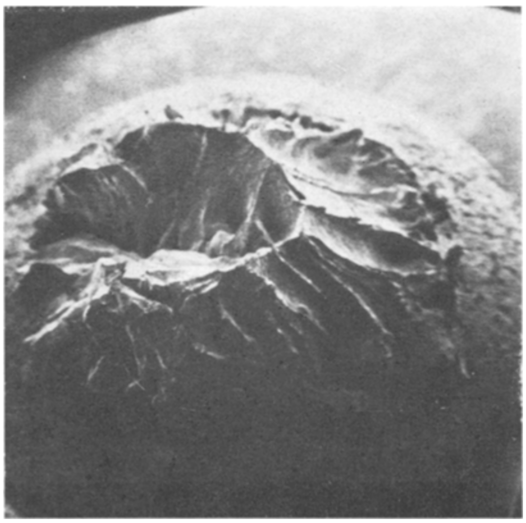

Figure 5 Scanning electron micrograph of the fracture surface of a pure iron specimen (notch 0.5 mm deep, 0.5 mm wide) which had been creep tested at 1100°C under an applied stress of 0.55 tons/in^2 at the notch (time to failure 790 min.; elongation 13.5%) (× 29).

It was noted that the cavities were more numerous as the amount of liquid copper phase increased. In some cases, whole grains near the notch area were isolated by the linking-up of cracks and liquid phase (fig. 2).

The final stages of the failure process occurred when surface cracks and cavities extended along the copper-rich phase and eventually linked-up (fig. 3).

Several of the observed features of embrittlement were similar to those reported by Williams and Singer [7] for the aluminium/liquid tin system.

3.1. Fractography

A scanning electron micrograph of the fracture surface of a copper-coated iron specimen, which had been stress-rupture tested at 1100°C is shown in fig. 4. The fracture surface is markedly different to the fracture surface of pure iron (fig. 5), which has the appearance of a ductile type fracture. Fig. 6 shows a large intercrystalline crack which has grown from the root of the notch. Intercrystalline triple point cracks which have formed in the central zone of the fracture surface are illustrated in fig. 7.

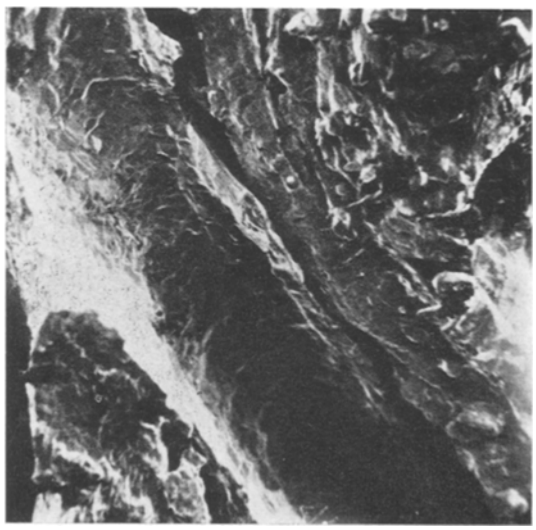

Figure 6 As fig. 5, showing a large intercrystalline crack which has grown from the root of the notch (× 290).

In contrast to these features, the copper embrittled iron specimen revealed dark spots (16 μm diameter) on the central fracture surface (fig. 8). Since these spots were not observed on the pure iron fracture surface (figs. 5-7), they are thought to be due to the presence of copper in the specimen.

During creep, copper films form along the grain boundaries of the iron. In subsequent cooling to room temperature, droplets of copper would be formed on the fracture surface because the copper films contract ("de-wet") towards the equilibrium shape. Similar, characteristic "dark spots" were observed by electron microscopy of the diffusion zone of grain boundaries penetrated by copper during creep (fig. 9) which again suggests that the spots were probably copper particles. Eborall and Gregory [8] observed a

Figure 7 As fig. 5, showing intercrystalline triple point cracks which have formed in the central zone of the fracture surface (× 134).

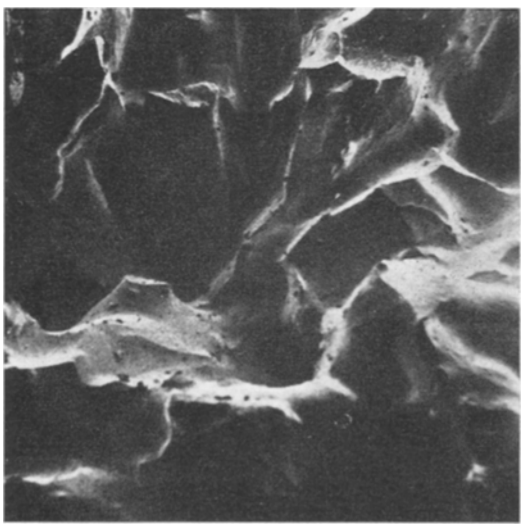

Figure 8 As fig. 4, showing dark spots on the central fracture surface (× 60).

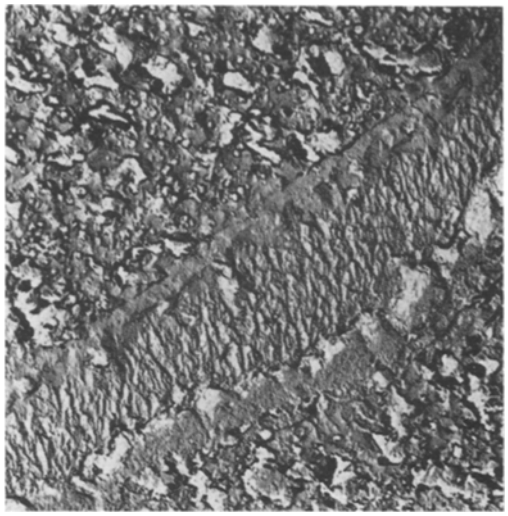

Figure 9 Iron with a 0.5 mm deep, 0.5 mm wide notch containing 0.29 notch depth of copper, creep tested at 1100°C under an applied stress of 0.55 tons/in^2 at the notch (creep strain 2.5%; creep time 134 min.). An electron micrograph of the diffusion zone of a grain boundary penetrated by copper during creep which shows characteristic "dark spots" (× 10000; Carbon Replica).

Figure 10 As fig. 4, showing that the form and shape of the "dark spots" closely resembles the characteristics of liquid droplets which have solidified on a metal surface (× 720).

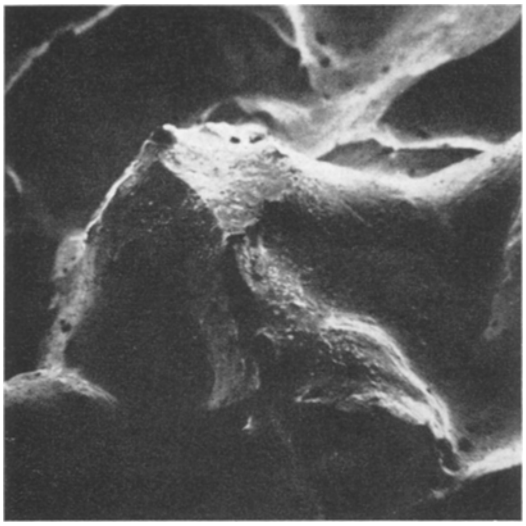

Figure 11 As fig. 4, showing the linking-up of an intercrystalline crack with particles of copper on the fracture surface (× 127).

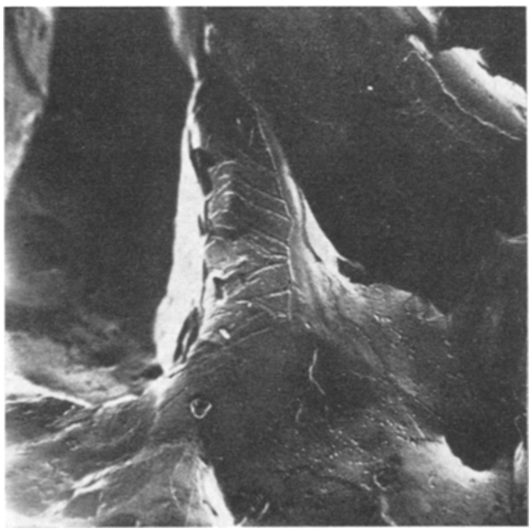

Figure 12 As fig. 4, showing wavy slip lines on the fracture surface (× 245).

similar de-wetting effect for liquid lead on a β-brass fracture surface.

At a higher magnification, a typical region of the fracture surface containing dark spots (fig. 10) strongly suggests that their form and shape closely resembles the characteristics of liquid particles which have solidified on a metal surface This evidence strongly supports the conclusion. that the "spots" are areas of solidified copper-rich phase.

A closer examination of the fracture surface of fig. 4 revealed that intercrystalline cracks did, in fact, link up with copper particles on the fracture surface (fig. 11). However, there was evidence of wavy slip lines on the fracture surface (fig. 12), which suggests that some plastic flow had taken place before fracture. At higher magnifications,

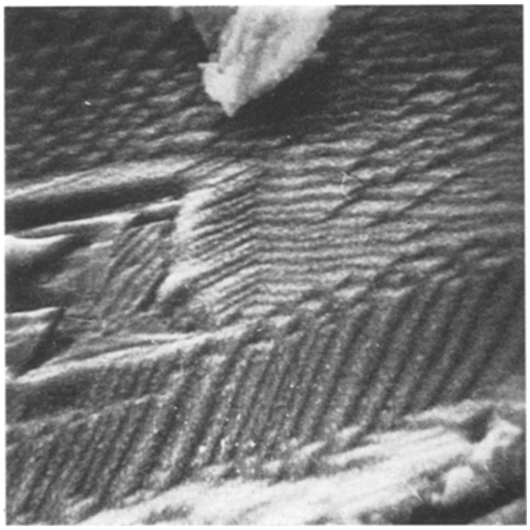

Figure 13 As fig. 4, showing a striation pattern for individual grains on the fracture surface, which resembles characteristic fatigue markings (× 7200).

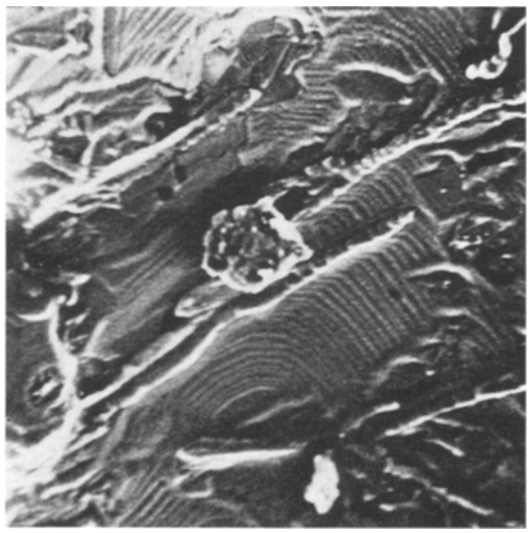

Figure 14 As fig. 13 (× 2550).

the fracture surface of copper-coated iron specimens showed an interesting striation pattern for individual grains (figs. 13 and 14), which resembled characteristic fatigue markings. These features were not observed at similar magnifications on the fracture surface of pure iron specimens. It is, therefore, suspected that their origin may be related to the presence of liquid copper rich phase during creep deformation. The striations could have formed either from the solidification front of the liquid copper or from a selective adsorption of copper atoms on certain crystallographic planes.

4. Conclusions

1. The observed embrittlement of iron was of the delayed failure type from diffusion-controlled grain boundary penetration by copper with accompanying extended surface notching.
2. Linking of surface cracks and internal cavities along copper-penetrated grain boundaries was the final failure mode.
3. The principal controlling processes for crack growth were the rate and amount of copper diffusion ahead of a growing crack and not the feed of liquid to the crack tip.

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