Observations of sub-micron cavities in a low alloy ferritic steel

The process of cavity nucleation at grain boundary particles has been studied extensively both theoretically and experimentally [1–4], and there is now substantial evidence in support of grain boundary particles acting as the main nucleating sites [2]. Grain boundary particles act as obstacles to grain boundary sliding, resulting in the formation of a stress concentration causing decohesion of the particle–matrix interface [3]. In order to study the nucleation process, it is necessary to use high resolution electron optical techniques. It is only recently with the application of such techniques that adequate nucleation studies have been made [2, 4, 5]. Dyson *et al.* [4] and Fleck *et al.* [2] using 1 MV transmission electron microscopy (TEM) have both observed sub-micron cavities, the latter clearly showing grain boundary particles to be the main sites for nucleation. The purpose of this short note is to provide evidence of preferential sub-micron cavity nucleation at grain boundary carbides in a low alloy ferritic steel, by using scanning electron microscopy (SEM) techniques.

Constant strain rate tests at 10^{-6} sec^{-1} were carried out on a 1.5% Cr–0.5% V low alloy ferritic steel containing 0.22% C, at 650° C under vacuum. More extensive information regarding the creep fracture behaviour of this alloy has been reported elsewhere [6]. The material was heat treated to give a tempered martensite structure. Examination (TEM) of carbon replicas revealed the structure to consist of M_7C_3 and M_4C_3 carbides distributed in the matrix and along the prior austenite grain boundaries. The average diameter of the M_7C_3 and M_4C_3 carbides at the grain boundaries was found to be 0.33 μm and 0.04 μm respectively. Specimens were tested to varying strains up to fracture. Tests stopped prior to fracture were subsequently fractured at –196° C, which resulted in a fracture face consisting of almost entirely intergranular facets. Fracture faces were examined by SEM enabling quantitative and qualitative information regarding cavity nucleation and growth to be obtained. Examination of the exposed grain boundary facets not only revealed the cavitation behaviour, but in some cases exposed the grain boundary carbide distribution present at the boundaries. Fig. 1 shows a SEM fractograph of a boundary facet taken from a specimen strained 1.0%. The grain boundary carbides present are aligned in one direction in the plane of the boundary. The size of the carbides is similar to the M_7C_3 carbides observed in the carbon replicas during TEM studies. In some instances the carbides have remained in the other half of the fracture face, i.e. A in Fig. 1. The observed alignment of grain boundary carbides in one direction in the boundary plane has also been reported in an Al–Cu alloy [7]. At some of the carbides in Fig. 1 small sub-micron cavities have nucleated (i.e. at B), these cavities are approximately 0.09 μm in radius. A number of other relevant points arise from Fig. 1; firstly the cavities have nucleated at the same side of the carbide particles, which would be expected if the stress concentrations were formed due to grain boundary sliding. Secondly, it appears that nucleation occurs by decohesion of the carbide–matrix interface, since the carbides in Fig. 1 show no signs of cracking. Finally, nucleation is a continuous process and occurs preferentially at grain boundary carbides. This is apparent since not all carbides have nucleated cavities. Fig. 2 shows a fractograph from a specimen strained to 1.5%. In this case the cavities have reached a far larger size and in some cases interlinking has occurred. As in Fig. 1, the grain boundary carbides are again mostly aligned in one direction in the boundary plane, and also not all the carbides have nucleated cavities. It is clear from Fig. 2 that during growth the cavities envelop a number of neighbouring

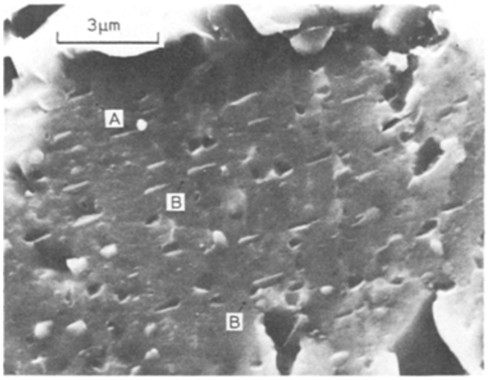

Figure 1 Sub-micron cavities nucleating at grain boundary carbides in 1.5% Cr–0.5% V low alloy steel, after 1.0% strain at 650° C.

carbides lying in the plane of the boundary; hence cavities tend to contain a number of carbides.

The minimum critical radius of a cavity is dependent on the applied stress through the equation $r = 2\gamma/\sigma$ [3], where r = cavity radius, γ = surface energy and σ = applied stress. There have been no cases reported [8] of cavities being observed having radii smaller than that given by the above equation, since below this value surface tension forces would be too strong to permit the cavity to remain. The minimum radius is not only dependent on the applied stress but also on the material, i.e. in Cu, Mg typical minimum radii of $\sim 0.3\,\mu$m, are observed whereas for high creep resistant alloys such as low alloy ferritic steels, the minimum radii can be as small as $0.01\,\mu$m. In the present case, taking $\gamma = 2\,\text{J}\,\text{m}^{-2}$ and $\sigma = 135\,\text{MN}\,\text{m}^{-2}$, then the minimum cavity radius is $0.015\,\mu$m. Hence, the sub-micron cavities observed in Fig. 1 have just grown to above the minimum radius.

In conclusion, the observations show that it is possible to observe sub-micron cavities using SEM techniques and that in the low alloy steel studied nucleation occurs continuously and preferentially at grain boundary carbides by a process of decohesion of the carbide–matrix interface.

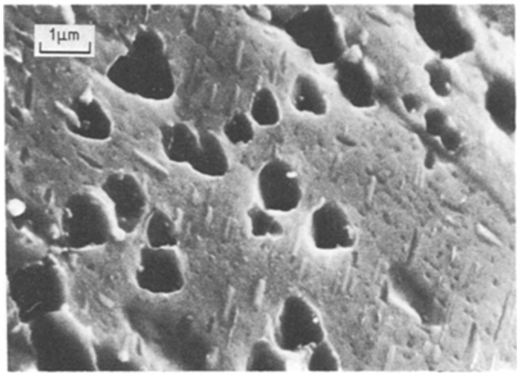

Figure 2 Grain boundary cavities enveloping carbides during growth in a 1.5% Cr–0.5% V low alloy steel after 1.5% strain at 650° C.

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Anodic oxidation of InP and the quaternary alloy $Ga_xIn_{1-x}As_yP_{1-y}$

The full potential of semiconductor materials in the fabrication of electronic devices based on planar configurations may only be realized if suitable methods are available to form insulating oxide layers of acceptable qualities on their surfaces. Early work had relied on gas phase oxidation [1] but recently, following the work of Spitzer *et al.* [2], Hasegawa *et al.* [3, 4] and Hasegawa and Hartnagel [5] on the anodic oxidation of GaAs, electrochemical techniques with high electrolyte stability, reliability of procedure free from severe contamination effects and excellent reproducibility have become important. Accordingly, in a solution consisting of 3 wt% aqueous solution of tartaric acid mixed with propylene glycol (PGT), glass-like oxide films of thickness up to about 8000 Å may be grown on n-type GaAs in a few minutes. Factors such as electrolyte composition, pH, anodization current density and surface illumination decide the thickness and conductivity of the layers formed. In this