The influence of primary precipitates on the tensile strength of unidirectionally solidified (Fe, Cr) – (Cr, Fe), C₃ *in-situ* grown composites containing 30 wt % Cr

J. VAN DEN BOOMGAARD, A. M. J. G. VAN RUN Philips Research Laboratories, Eindhoven, Netherlands

The influence is investigated of metal dendrites and primary carbide needles on the tensile strength of the (Fe, Cr) – (Cr, Fe)₇C₃ *insitu* grown composite cont-aining 30 wt % Cr. It is found that these irregularities in the aligned structure diminish the tensile strength of the *in-situ* composite both at room temperature and at 900°C. The maxima in the tensile strength versus composition curves occur at different compositions for these temperatures. A possible explanation of this behaviour is given.

1. Introduction

Faults in *in-situ* grown composites, such as grain boundaries, banding and primary precipitates, can have a pronounced effect on the strength properties. These faults may induce fractures due to inhomogeneous distribution of the solid phases, misalignment of primary precipitates, absence of one of the phases in the bands etc.

In this paper the results are reported of an investigation into the influence of primary precipitates on the tensile strength of the *in-situ* grown (Fe, Cr) – (Cr, Fe)₇C₃ composite. This composite consists of hexagonal (Cr, Fe)₇C₃ rods, with the rod axes parallel to the crystallographic *c*-axis, in a γ (fcc) or an α (bcc) matrix of Fe and Cr.

In the ternary Fe-Cr-C system a whole curve exists, giving the compositions of samples, exhibiting aligned eutectic structures after unidirectional solidification in steady state conditions (curve AB in Fig. 1). Samples with composition B exhibit the smallest diameter of the carbide rods, when compared with similarly prepared samples on AB. They also show a high, reproducible tensile strength [1]. Therefore bars with compositions close to B were selected for an investigation of the influence of metal dendrites and primary carbide needles on the tensile strength of the composite.

2. Experimental

Bars of composition (70-*x*) wt % Fe 30 wt % Cr



Figure 1 Curve in the ternary Fe-Cr-C system showing the compositions of (Fe, Cr) – $(Cr, Fe)_7C_3$ composites with regular structures after unidirectional solidification under steady state conditions.

and x wt % C were prepared by unidirectional solidification at a rate of 30 cm h⁻¹, using the floating zone technique in an atmosphere of H₂. The purity of the starting materials was the same as that described earlier [1]. The carbon content x was varied from 2.7 to 3.3 wt %, the limiting values being given by the points C and D in Fig. 1. B corresponds to ~ 2.9 wt % carbon.

The tensile strengths of the bars were measured both at room temperature and at 900°C using a method which will be described in a forthcoming paper [2].

3. Results

The results obtained for the dependence of the tensile strength on the composition at both temperatures are given in Tables I and II, and are plotted in Fig. 2 and on a normalized scale in Fig. 3. Figs. 4, 5 and 6 show micrographs of the

TABLE I Tensile strength of the unidirectionally solidified composite material (Fe, Cr) – (Cr, Fe)₇C₃ containing 30 wt % Cr and x wt % C at room temperature in air.

$\frac{x}{(\text{wt }\% \text{ C})}$	Number of samples	$\sigma_{\rm B}$ (kgmm ⁻²)	
		Average value	Maximum value
2.7	5	162	175
2.8	7	184	188
2.9	6	191	210
3.0	7	195	207
3.1	5	158	175
3.3	3	82	92

TABLE II Tensile strength of the unidirectionally solidified composite material (Fe, Cr) – (Cr, Fe)₇C₃ containing 30 wt % Cr and x wt % C at 900°C in air.

x (wt % C)	Number of samples	σ _B (kg mm ⁻²)	
		Average value	Maximum value
2.7	3	17.1	21.0
2.8	3	24.2	27.8
2.9	3	30.5	30.9
3.0	3	31.3	33.9
3.1	3	32.6	33.6
3.3.	3	15.7	24.5

longitudinal sections in the neighbourhood of the fracture surface for bars containing 2.8, 2.9 and 3.0 wt % C respectively. These show the presence of metal dendrites (Fig. 4), the absence of primary phases (Fig. 5) and the presence of primary carbide rods (Fig. 6). In Fig. 4 it can be seen that a crack has started at a surface in which a metal dendrite is present and Fig. 6 shows that primary carbide rods act as starting points for cracks.

The carbide rods in the samples with regular structures are several hundred microns long and the average thickness is $\simeq 2 \ \mu m$.

Both types of primary phases decrease the tensile strength of the material (Fig. 2). From the normalized curves in Fig. 3 it is evident that the influence of the primary phases is different at both temperatures. The maximum value of the



Figure 2 Average tensile strength, $\sigma_{B_{av}}$, of the composite material (Fe, Cr) – (Cr, Fe)₇C₃ composites with 30 wt % Cr as a function of the carbon content x at 25°C and at 900°C in air.



Figure 3 The curves of the average tensile strengths, $\sigma_{B_{av}}$, of Fig. 2 normalized to their maximum values.

tensile strength versus the C content at room temperature is found at 2.92 ± 0.03 wt % C whereas at 900°C it is situated at 3.04 ± 0.03 wt % C.

4. Discussion

4.1. The influence of growth rate and temperature gradient



Figure 4 The composite with metal dendrites. Longitudinal section in the neighbourhood of the fracture surface for a sample containing 2.8 wt % C tested at 900°C in air (magnification \times 200).



Figure 5 Micrograph of a longitudinal section in the neighbourhood of the fracture surface for a sample containing 2.9 wt % C tested at 900°C in air (magnification \times 200). Primary phases are absent.

Curve AB in Fig. 1 represents the curve of compositions corresponding to aligned structures of $(Cr, Fe)_7C_3$ rods in a (Fe, Cr, C) matrix, to be obtained by unidirectional solidification, provided the temperature gradient (G) at the solidification front is sufficiently large to prevent constitutional supercooling. At a growth rate, R,

and a temperature gradient, G, regular structures can also be obtained in a small region around AB, the size of which will depend on G/R. This is similar to the phenomenon of regular offeutectic growth in a binary eutectic system [3]. It means that in the case of regular growth at the given chromium content the carbon content may



Figure 6 The composite with primary carbide rods. Longitudinal section in the neighbourhood of the fracture surface for a sample containing 3.0 wt % tested at 900°C in air (magnification \times 200).

vary in a small region about the composition B, without the occurrence of primary phases (Figs. 2 and 3). Because a variation of the carbon content at a constant chromium content corresponds to a variation in the volume fraction of the carbide phase in the same sense, regular structures are only found in a small region of carbide volume fraction depending only on G and R (2.9 wt % C corresponds to about 33 vol % carbide rods).

Outside this region irregular structures are found; primary carbide rods at higher volume fractions of the carbide phase (higher C contents) and metal dendrites at a lower volume fraction of the carbide phase (lower C contents). We observed that the alignment of the primary carbide rods is better at higher solidification rates, which is also found by other authors [4, 5]. In our experimental conditions we found an increase in misalignment of these rods with an increasing number of primary rods at a fixed growth rate.

Because 30 cm h^{-1} is a rather high growth rate we may expect that, if only few primary rods are present, most of them will be quite well oriented.

4.2. The mechanical properties of the two phases and of the composite.

The matrix consists of Fe, Cr and a small amount of C, i.e., the matrix is a low-carbon chromium steel, exhibiting a rather high tensile strength at room temperature, but a low ductility. At 900° C, on the contrary, it has a low tensile strength but it is very ductile. As a consequence the matrix will be very notch-sensitive at room temperature but not at 900° C.

Because the carbide is a refractory material the tensile strength and the (low) ductility of the carbide rods are almost independent of temperature. The tensile strength depends only on the rod diameter and increases as the diameter is decreased.

The tensile strength, $\sigma_{\rm B}$, of the composite increases with increasing volume fraction of the rods. This is schematically indicated in Fig. 7 by the dashed curves (a) for room temperature and (b) for 900°C, which generally are not straight lines.

In the *in-situ* grown composite, grown at a fixed R and G, only a small region of aligned composites is possible around 33 vol % of carbide fibres [1]. In Fig. 7 PQ and P'Q' would represent the tensile strength of the composite at room temperature and at 900°C in the region without primary phases if no other influences were present. The great difference in $\sigma_{\rm B}$ of the composite at the two temperatures is caused by the difference in mechanical properties of the matrix (see above). For the same reason the slope of (b) may be expected to be steeper than the slope of (a).

Samples with carbide volume fractions below



Figure 7 Schematic drawing of the tensile strength σ_B at room temperature and at 900°C accounting for the influences of primary phases.

this region of aligned structures exhibit dendrites of the matrix phase. These dendrites cause an extra decrease of the tensile strength compared with the extrapolated PQ and P'Q' (PV and P'V' respectively).

Samples with carbide volume fractions above the region of aligned structures exhibit primary carbide rods. A number of these rods are fairly well aligned, a fraction of them are not. This fraction becomes greater the greater the volume fraction of the carbide phase at fixed growth rate and temperature gradient. The thick aligned primary rods may contribute to the tensile strength of the composite, although less than the thin eutectic ones, the badly aligned ones will not. Therefore, as in the case of metal dendrites, the $\sigma_{\rm B}$ of samples with primary carbide rods will be lower than the values obtained by extrapolation of PQ and P'Q' (Q'S and Q'S respectively). However, the decrease of $\sigma_{\rm B}$ will be different at the two temperatures as a result of the behaviour of the matrix. Some hexagonal carbide rods easily cleave parallel to their *c*-axis. Because the rod axis is parallel to the *c*-axis a primary carbide rod situated perpendicular to the growth direction of a sample may be split along the rod axis by the force transferred to it by the matrix. At room temperature such a cleaved carbide rod acts as a severe notch in the brittle matrix. In Fig. 8 a part of a fracture surface is shown containing only one such longitudinally split

primary carbide rod in its centre perpendicular to the growth direction of the sample. The radial pattern of the fracture surface indicates that the cleaved primary rod acted a fracture source. At room temperature the tensile strength of this sample was 148 kgmm⁻² instead of 200 kgmm⁻² found for structures without primary phases, i.e., a decrease of 25% from which it may be concluded that the presence of some misaligned primary carbide rods may have a deletereous effect on the tensile strength of the composite at room temperature. As small fluctuations in composition of the composite within the region of aligned structures may cause primary carbide rods, especially if the composition is situated close to the boundary with the region in which primary carbide rods are always present, a decrease of $\sigma_{\rm B}$ may already start within this region of aligned structures. As a consequence the maximum of $\sigma_{\rm B}$ may be situated within this region (M_1 in Fig. 7).

At 900°C, however, the matrix is not very notch-sensitive and the occurrence of badly aligned primary carbide rods will not have such a deletereous effect, whereas the aligned ones may contribute to the tensile strength of the sample. As a consequence the tensile strength may still increase after the passage of the boundary between the region of aligned structures and the region in which primary carbide rods appear, although less than indicated by Q'S'. Upon a



Figure 8 The central part of a fracture surface showing the fracture source: one primary carbide rod perpendicular to the growth direction of the sample (magnification \times 180).

further increase of the volume fraction of the carbide phase the number of thick primary rods and especially the number of misaligned ones will increase so much that $\sigma_{\rm B}$ will decrease again. As a consequence the maximum value of $\sigma_{\rm B}$ may be situated in the region of primary carbide rods (M₂ in Fig. 7). This shift of the maximum to higher C-contents at higher temperatures will be greater the steeper the slope of (b). The experimental results represented in Fig. 2 are qualitatively in agreement with the curves VM₁W and V'M₂W' of Fig. 7.

5. Conclusion

Metal dendrites as well as primary carbide rods diminish the tensile strength of the *in-situ* grown composite with respect to the values it would have if only aligned structures occurred at the given composition.

Due to the difference in ductility of the matrix the maximum of $\sigma_{\rm B}$ at room temperature is situated within the regular region, while at 900°C the maximum is shifted to the region where primary carbide rods are present.

Acknowledgements

Thanks are due to Mr J. Liebe for careful preparation of the samples, to Mr J. Scholing for the tensile tests at room temperature and to Mr W. Janssen for his aid in the tensile tests at 900° C.

References

- 1. J. VAN DEN BOOMGAARD and L. R. WOLFF, J. Cryst. Growth **15** (1972) 11.
- 2. A. M. J. G. VAN RUN, to be published.
- 3. F. R. MOLLARD and M. C. FLEMINGS, *Trans. Met.* Soc. AIME 239 (1967) 526.
- 4. F. D. LEMKEY, private communication.
- 5. P. R. SAHM, private communication.

Received 30 October and accepted 20 November 1972.