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TEM INVESTIGATIONS OF WC-Co ALLOYS AFTER CREEP EXPERIMENTS

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Résumé - Des alliages carbure de tungstène-cobalt ont été déformés par compression ou par flexion trois points dans un domaine de température 1000-1350°C et dans un domaine de contrainte 30-1000 MPa. Dans ces conditions, l'exposant des contraintes n des alliages WC-Co est fonction uniquement du pourcentage volumique en cobalt et tend vers n = 1 pour le carbure pur. L'énergie d'activation apparente est 550 kJ.mole⁻¹. Des observations en M.E.T. sur des échantillons de carbure pur déformé à 1450°C montrent une déformation intragranulaire importante. L'analyse de ces défauts a été réalisée. De plus, une étude précise a montré un comportement différent entre les joints de grains en coïncidence (Σ = 2, 4, 13a) et les joints de grains généraux. Ces analyses à l'aide des données macroscopiques permettent de proposer un modèle pour la déformation des composites carbure de tungstène-cobalt.

Abstract - Carbide tungsten cobalt alloys were deformed in compression or in three point bending in a temperature range 1000-1350°C and in a stress domain 30-1000MPa. In these conditions, the stress exponent n of WC-Co alloys is a function of only the cobalt volumic ratio and tends towards n = 1 for pure carbide. The apparent activation energy is 550 kJ mole⁻¹. T.E.M. investigations on pure carbide deformed at 1450°C show an extensive intragranular deformation. Analysis of these defects have been performed. Moreover an accurate study has shown a different behaviour between coincidence grain boundaries (Σ = 2, 4, 13a) and general grain boundaries. These data and macroscopic results allow to propose a model for the deformation of carbide tungsten - cobalt composites.

I - INTRODUCTION

Up to recently the high temperature deformation behaviour has been mainly investigated at temperatures less than 1000°C. Only Gottschall, Williams and Ward /1/ made measurements at temperature up to 1400°C. A systematic study of the creep of WC-Co in the temperature range 1000-1350°C has been started in our laboratory /2/. Thus the general creep behaviour of the composite and the influence of the microstructure on its parameters have been determined. Due to the lack of informations on the dislocation structures after deformation, it has not been possible to propose a creep mechanism. The reason is the complexity of the defect structure of the carbide in the as-sintered state /1,3/.

The aim of this work is to get accurate informations on the dislocation structures after high temperature deformation. To do this we have previously annealed as-sintered samples at 1700°C ; the defect structure thus disappears /4/. These samples will be denoted as pure carbide in the remaining of this paper. Thin slices of these samples deformed at high temperature have been observed in T.E.M.. The expected results are correlated with macroscopic data in order to propose a microstructural creep model of the WC-Co composites.
II - EXPERIMENTAL PROCEDURE

Samples with different granulometry ($D_{WC} = 0.7$, 1.1, 2.2 $\mu$m) and different cobalt volumic ratio ($V_{(Co)} = 5-37\%$) were deformed in compression or in three point bending under vacuum. The creep experiments were performed in the temperature range 1000-1350°C and in a stress domain 30-1000 MPa. For electron microscopy investigations, specimens ($5 \times 5 \times 5 \text{ mm}^3$) were spark-cut from sintered WC-Co alloys (3 wt % Co). The samples had been annealed at 1700°C for four hours. During this treatment, the cobalt phase evaporated. These samples have been deformed in compression at 1450°C and cooled under load with a strain value of 15 %. Thin slices were spark machined down to a thickness of 80$\mu$m and 3 mm in diameter. These discs were then ion milled for electron microscope investigations. Jeol 100CX and 200CX microscopes were used.

III - CREEP BEHAVIOUR OF WC-Co MATERIALS

Results of the creep experiments have been plotted in a log$e$-log $\sigma$ diagram. The resulting curves have a sigmoidal shape with three stages possessing a different stress exponent. The intermediate stage (stage II) has been particularly studied. In stage II, the stress exponent, $n$, is shown to depend only on the cobalt volumic ratio, tending to a value of 1 as the cobalt content tends to zero independently with the carbide grain size (Fig. 1). The influence of the latter appears however in the strain rate expression as: $\dot{\varepsilon} \propto \sigma^{n} D_{WG}^{-2}$, where $D_{WG}$ is the mean carbide grain size. The value of the apparent activation energy $Q_{A}$ in stage II is 550 kj mole$^{-1}$ (Fig.2). A same value for $Q_{A}$ has been found for all the batches used in this study. This value is high when compared with self-diffusion energy of the cobalt. On the other hand the deformation of the material is obtained at high stress levels. It appears thus that the deformation of the composite is controlled by the carbide phase. Observations of deformed materials had been made using a scanning electron microscope. The specimen had been polished, then scratched and finally deformed to various levels of deformation. Grain boundary sliding of the carbide grains had been observed either as individual crystals or as groups of crystals /2/.

IV - TEM OBSERVATIONS AND ANALYSIS IN PURE WC DEFORMED AT 1450°C

TEM observations of the carbide phase after high temperature deformation reveal an extensive intragranular deformation. Dislocation tangles, extended dislocations and

![Graph 1](image1.png)

**Fig. 1** - Stress exponent as a function of cobalt volume ratio.

![Graph 2](image2.png)

**Fig. 2** - Creep activation energies for various materials:
- $V_{(Co)} = 5\%$ A103 ($D_{WC} = 2.2 \mu$m), H03T ($D_{WC} = 0.8 \mu$m), A2003 ($D_{WC} = 0.7 \mu$m) (three point bending)
- $V_{(Co)} = 37\%$ A2025 ($D_{WC} = 0.7 \mu$m) (compression).
a large number of low angle grain boundaries are the main defects. Low angle grain boundaries have already been studied in details \cite{4,5,6}. The majority of them observed in WC are due to the high temperature deformation treatment. Different types of subgrains have been found; they are mainly tilt subgrain boundaries formed by a single family of perfect dislocations \(1/3 \langle 11\bar{2}0\rangle (I), \langle 0001\rangle (II)\) and \(1/3 \langle 11\bar{2}3\rangle (III)\). They can result from dislocation sources in the planes \((0001)\) or \((10\bar{1}0)\) for types (I) and (II) or pyramidal \((11\bar{2}1)\) planes for type (III) and climb processes.

As a comparison, defects resulting from plastic deformation at room temperature are predominantly extended dislocations \(1/6 \langle 11\bar{2}3\rangle\) which are glissile configurations in the \((1\bar{1}00)\) planes \cite{3,7,8}. Some isolated and dissociated dislocations \(1/6 \langle 11\bar{2}3\rangle\) have been also found after high temperature deformation. They clearly show a recombination over a part of their line due to climb processes; A detailed analysis of such a dislocation is presented on Fig. 3. Fig. 3a) is a weak beam image obtained with the \(0\bar{1}1\bar{1}\) reflection. The dislocation is dissociated in the prismatic \((10\bar{1}0)\) plane \((12.5 \text{ nm})\) in the part denoted \(L_1\) and not dissociated in the part \(L_2\). In Fig. 3b) the \((10\bar{1}0)\) dissociation plane is nearly parallel to the electron beam; therefore the two dislocation lines are superimposed in the part \(L_2\). Fig. 3c) is the stereographic projection of the precedent case. The Burgers vector, the dislocation line directions \(L_1\) and \(L_2\) and the dissociation \((10\bar{1}0)\) plane are indicated on the drawing.

Systematic observations of grain boundaries have been performed after high temperature deformation on coincidence and general grain boundaries. It appears that many coincidence grain boundaries frequently observed in the material \((3 \, (Z = 2, 4, 13a)\) reveal the presence of a number of dislocations in their boundary plane. On the contrary they are not observed in general grain boundaries. A detailed analysis of dislocations in a \(Z = 2\) grain boundary is presented on Fig. 4. Four grains (denoted

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**Fig. 3** - Climb of a dissociated dislocation: a) weak beam image obtained with \(g = 0\bar{1}1\bar{1}\). The part denoted \(L_1\) of the dislocation line is dissociated; A recombination of the dislocation line is observed in the part \(L_2\). The dislocation is locally dissociated in C. b) Bright field image obtained with \(g = 10\bar{1}1\). The two partial dislocations are superimposed. c) Stereographic projection. The dislocation line directions \(L_1\) and \(L_2\), the Burgers vector and the dissociation plane are indicated.
1, 2, 3 and 4) are imaged in Fig. 4.a. A characterization of their orientation has been performed. \( \Sigma = 2 \) grain boundaries have been found between 1 - 2 and 2 - 3 grains and grain boundaries between 1 - 4 and 3 - 4 are in a random orientation. Interactions between subgrains (denoted S - J on Fig. 4.a) and grain boundaries can be also seen. Only dislocations in the \( \Sigma = 2 \) grain boundary 1 - 2 have been analyzed. A magnified image of these dislocations is presented on Fig. 4.b. The stereographic projection is presented on Fig. 4.c. The Burgers vector of these dislocations 1/3 [1213] has a component out of the (1100) boundary plane (Fig. 4.c). Therefore the movement of these grain boundary structural dislocations (G.B.S.D.) involves glide and climb processes.

These observations can be related to previous studies (9, 10, 11) concerning the grain boundary sliding behaviour and dislocation structures; it has been proved that sliding of coincidence grain boundaries is difficult and slide hardening occurs. Dislocations are consequently observed. These observations are qualitatively in agreement with the present investigation. They can be explained by a dislocation model of sliding (12) where absorption of lattice dislocations in coincidence grain boundaries is more difficult than in general grain boundaries. If a climb process is involved for the movement of G.B.S.D., the amount of sliding depends on the grain boundary structure. The grain boundary diffusion and in consequence the climb rate are more difficult in coincidence grain boundaries than in general grain boundaries.

V - MICROSTRUCTURAL MODEL OF THE WC-Co COMPOSITES

The correlation between the previous results and the M.E.B. and T.E.M. observations of deformed samples allows one to propose two simultaneous mechanisms acting in the carbide phase during deformation:

- a grain boundary sliding of "isolated" crystals whose grain boundaries are interphase boundaries or random grain boundaries, giving a stress exponent of \( n = 1 \).

![Fig. 4 - Analysis of dislocations in a \( \Sigma = 2 \) grain boundary](image)

a) grain boundaries between 1 - 2 and 2 - 3 grains are \( \Sigma = 2 \) grain boundaries. Dislocations are imaged in their boundary planes. Grain boundaries between 1 - 4 and 3 - 4 grains are general grain boundaries. Frequent interactions between subgrains (denoted S - J) and \( \Sigma = 2 \) grain boundaries are observed.

b) Magnification of the grain boundary 1 - 2.

c) Stereographic projection corresponding to the grain boundary 1 - 2.
- an intragranular deformation of chains of crystals linked by coincidence grain boundaries. This mechanism is described by a power law creep \( n > 1 \). The observed value of \( n \) is a function of the relative number of these grain boundaries. Their ratio depends on the cobalt volumic ratio and seems to be a consequence of the sintering process.

A schematic representation of the microstructural model is presented in figure 5 where the three elements of the microstructure are indicated: cobalt crystals blue circles; chains of carbide crystals linked by coincidence grain boundaries green circles and "isolated" carbide grains orange squares. Sliding of "isolated" crystals induces the intragranular deformation of the adjacent crystal chains. Both mechanisms are accommodated by intragranular dislocation movement (glide and climb). This explains the constancy of the apparent activation energy over the whole domain of cobalt volumic ratio.

![Fig. 5 - Schematic representation of the microstructure of WC-Co composites.](image)

VI - CONCLUSION

Detailed microscopic investigations correlated with high temperature creep results show that WC-Co composites have a very specific microstructure which influences strongly their mechanical behaviour.

REFERENCES