JOURNAL OF MATERIALS SCIENCE 26 (1991) 2985–2990

Thermal expansion stress in a metallic matrix composite: *in situ* TEM observations

JEAN-PAUL COTTU, JEAN-JACQUES COUDERC
Laboratoire de Physique des Solides associé au C.N.R.S. (U.A. 74), U.P.S. et I.N.S.A., Avenue de Rangueil, 31 077 Toulouse Cedex, France

A metallic matrix composite material (2024-type aluminium alloy reinforced with 25 vol% SiC whiskers) was *in situ* thermally cycled in a transmission electron microscope. Dislocation movements were observed between 180 and 400 °C. The local shear stress due to the difference between the thermal expansion coefficient of matrix and fibres is estimated from the radius of curvature of mobile dislocations. The experimental values of the internal stress are discussed.

1. Introduction

The development of metallic matrix composite materials (MMC) which are specially devoted to applications in the medium temperature range, is closely related to the resolution of several problems. Among these, the differences between the thermal expansion coefficient of the matrix and the fibres are accountable for internal stresses and thermal fatigue with various consequences.

Observation by transmission electron microscopy (TEM) of such materials *in situ* thermally cycled has previously been performed. It shows the formation of very dense dislocation networks in the matrix areas located near the fibre/matrix interfaces [1, 2].

Plastification of the matrix owing to differential expansion has been recently studied by Dumant *et al.* [3] to help to understand the thermal expansion curves of MMC with an aluminium matrix and carbon fibre reinforcement.

This work gives an estimation of the residual stresses in the matrix depending on the material composition and the thermal treatment conditions. Our purpose was to measure directly the stress inside the matrix at several temperatures from dislocation movement observations. This paper presents our first results in this field.

2. Experimental procedure

The composition of the MMC used was analysed by powder metallurgy. The matrix was a 2024-type aluminium alloy, reinforced with 25 vol% SiC whiskers. In order to lower the dislocation density, so making TEM observation easier, the material annealed for 1 h at 400 °C and then slowly cooled to room temperature.

After mechanically grinding down to 100 μm thickness, the sample was thinned by ion milling. *In situ*

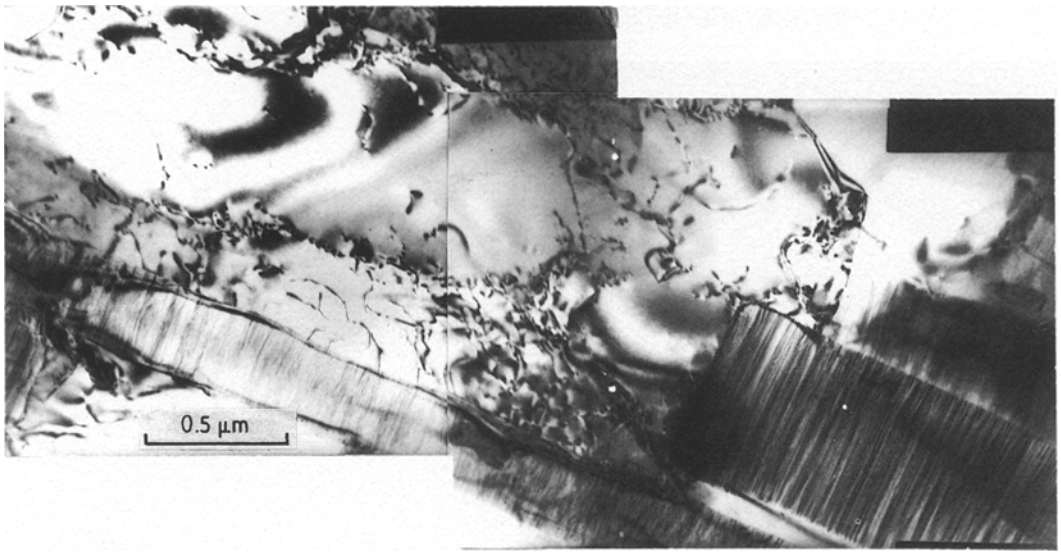

Figure 1 Bright-field electron micrograph. Characteristic heterogeneous dislocation distribution in the matrix; SiC whiskers are easily identified by their high stacking fault density.

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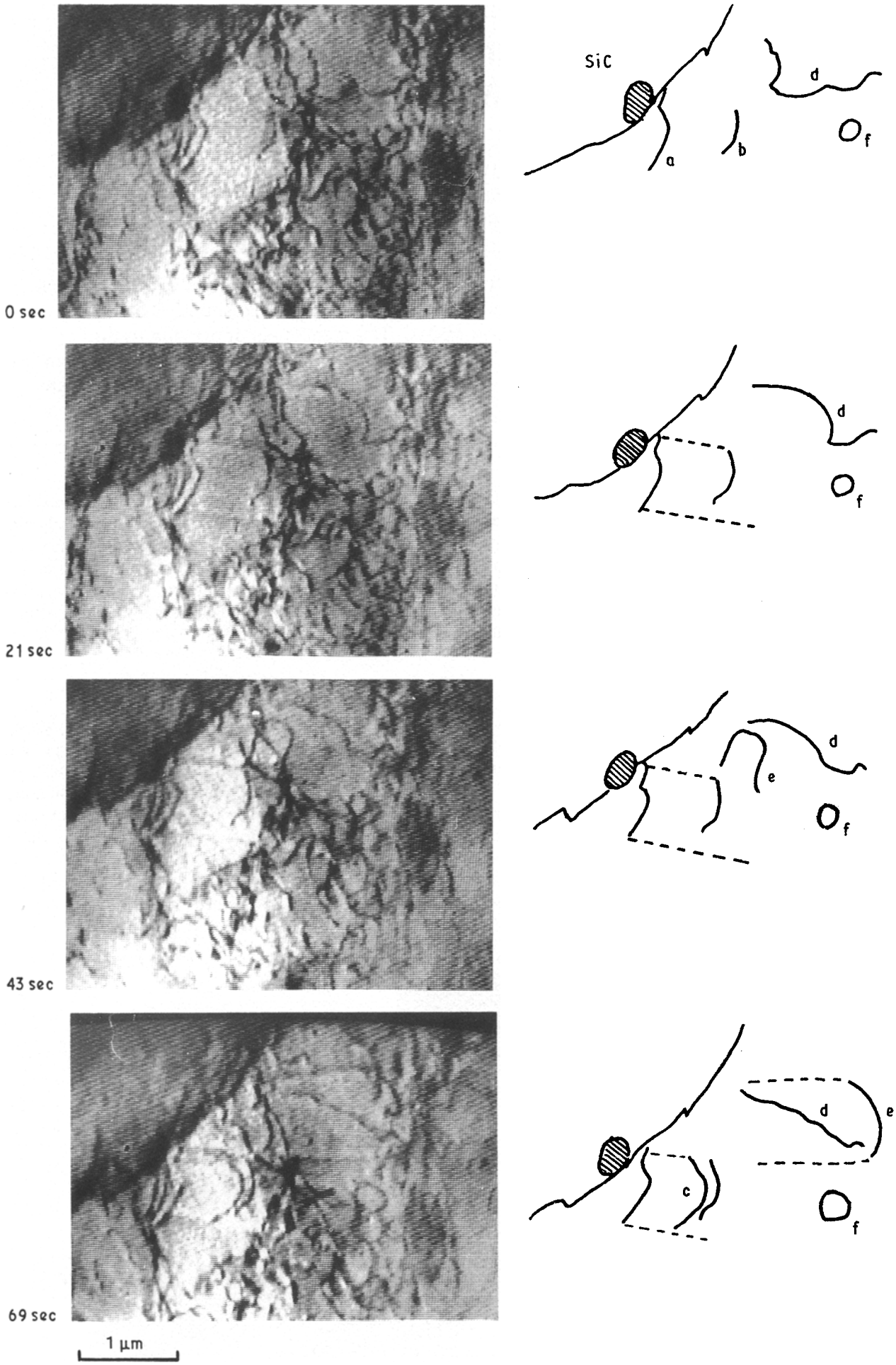


Figure 2 Sequence of in *situ* electron micrographs at 200 °C: sudden glide of dislocations c, d and e. Foil plane (2 1 3); glide plane (1 $\bar{1}$ 1); Burgers vector $\boldsymbol{b} = 1/2\ [1\,0\,\bar{1}]$.

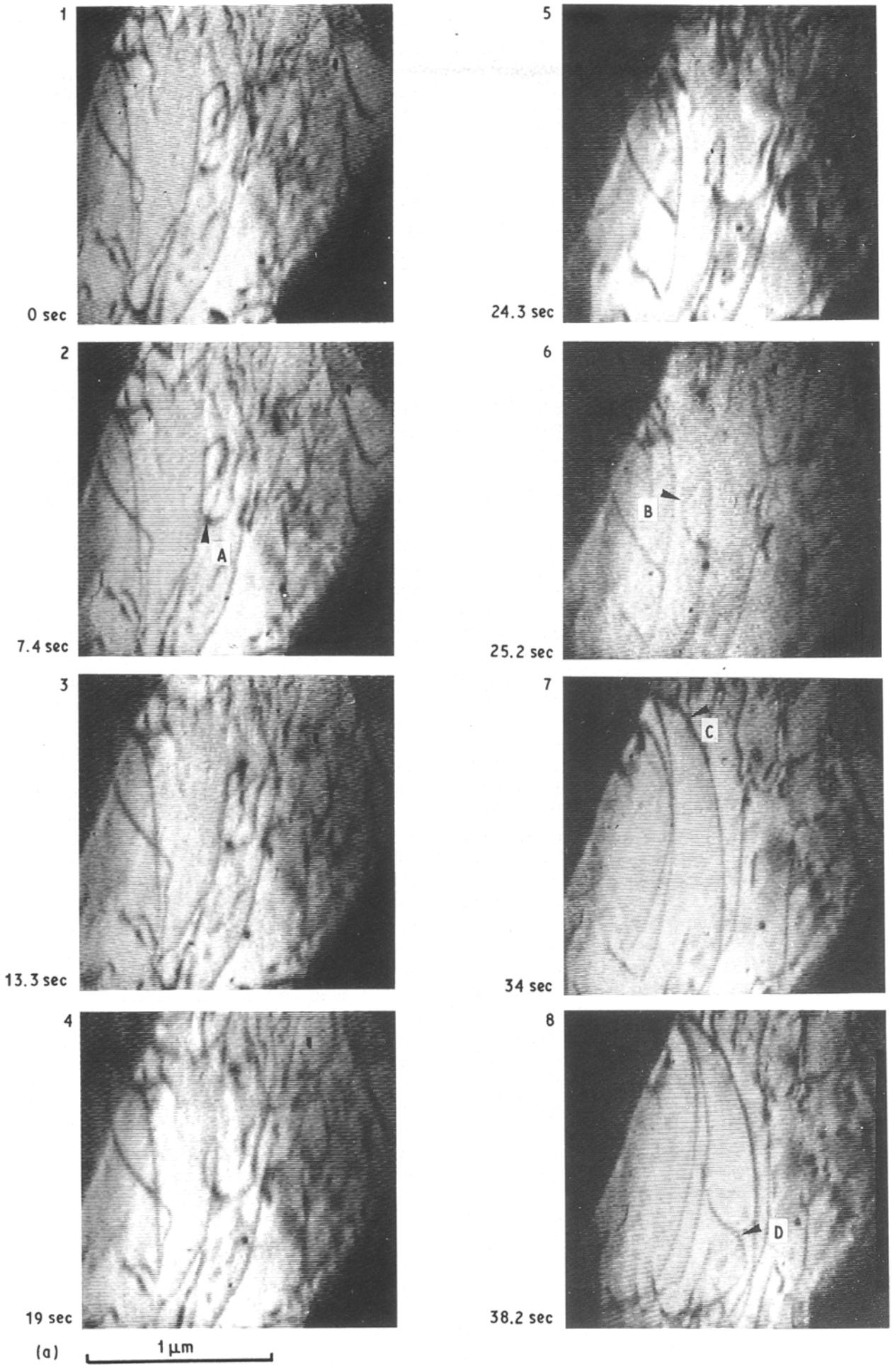


Figure 3 (a) A sequence of *in situ* electron micrographs: development of a dislocation loop in the centre of the observed area. Film plane $\simeq (0\,1\,1)$; glide plane $(1\,1\,1)$; Burgers vector $b = 1/2\,[0\,1\,\bar{1}]$. Stresses are computed from dislocation curvature at A, B, C and D. (b) Schematic representation of successive positions of the dislocation loop.

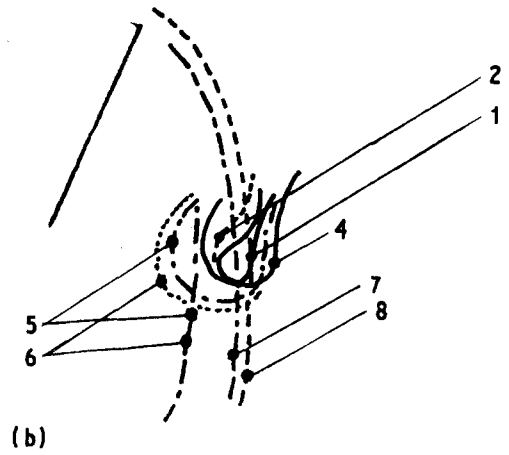


Figure 3 Continued.

heating experiments were performed on a Jeol 200 CX electron microscope (accelerating voltage 200 kV) equipped with a heating holder.

3. Results

The initial dislocation density was not uniform in the matrix zones located between the SiC whiskers: in spite of the slow cooling after annealing, the dislocation density was larger in the vicinity of matrix/fibre interfaces and fibre ends than in the centre of the interfibre space (Fig. 1). Tangled dislocations were observed, often pinned by precipitates.

During the first heating up to 200 °C, some dislocations were produced, generally from the matrix/fibre interfaces, between 180 and 200 °C. Dislocation movement, as shown in Fig. 2, occurred very suddenly. Glide traces implied a $(1\,\bar{1}\,1)$ slip plane making an angle of 52° with the thin foil surface (2 1 3). Cooling to room temperature did not lead to reverse movement of dislocations. Heating again at the same temperature did not change the initial dislocation configuration, and it was necessary to raise the maximum heating temperature higher than 200 °C in order to observe further dislocation glides in the same area. The video sequence shown in Fig. 3 was recorded on reheating the same foil up to 300 °C. The observed area was a matrix zone located between two SiC whiskers at a distance of about 1 μm, i.e. the mean distance between fibres. Several dislocation movements were observed and characterized. The glide plane of the dislocation loop emitted at A is (1 1 1), making an angle of 35° with the foil surface. The Burgers vector of this loop, $\boldsymbol{b} = a/2\ [0\,1\,\bar{1}]$ has been determined by the classical extinction rules. Then it was possible to measure the actual radius of curvature, R, of the dislocation segments in their glide plane. The local shear stress, τ, applied to the matrix was derived from the Orowan equation

$$\tau = \frac{\alpha \mu \boldsymbol{b}}{R}$$

where μ is the shear modulus; $\alpha \simeq 0.5$ to 0.8: $\boldsymbol{b}$ is the Burgers vector length.

Such computations made for the mobile dislocations visible in Fig. 3 gave the stress values at A, B, C and D: $\tau_A = 50$ MPa; $\tau_B = 35$ MPa; $\tau_C = 20$ MPa; $\tau_D = 28$ MPa.

In situ thermal cycling experiments were performed between 150 and 400 °C on a similar sample to that above. Fig. 4 shows dislocation glide in direct and opposite directions, on heating and cooling, during one of three successive cycles. From these figures, it is worth noting the difference between dislocation configuration before and after cycling (Fig. 1). The latter deformation test temperature was indeed a rather high one for an aluminium alloy.

4. Discussion

The presence of high dislocation density zones near the fibre/matrix interfaces had previously been reported [1]. These dislocations display the plastic deformation induced by differential thermal expansion between the fibres and matrix on cooling, even slowly.

Dislocation glide observed at 200 °C originates from compression stresses in the matrix, the thermal expansion of which is limited by that of the fibres. The glide is jerky and we have often observed such a behaviour during *in situ* deformation experiments performed at room temperature on aluminium alloys, after various thermal treatments [4]. It can be explained by the fact that in these precipitation hardenable alloys, dislocations are initially locked by solute atoms and by precipitates either migrating toward dislocations or gathering on them during the cooling treatment. Then dislocation glide needs a relatively high unlocking stress which is relaxed as soon as plastic deformation sets in. Moreover, other precipitates can stop dislocation glide. In these alloys stress computation is difficult at this low temperature, because the first stage of glide is not recordable. On cooling, the yield stress of the matrix increases and the differential expansion stress can promote neither reverse glide, nor de-anchoring of dislocations.

In order to observe again dislocation movement, it is necessary to perform the second test at a temperature higher than 200 °C; this fact could simply originate from work hardening resulting from the first test, but more probably precipitate hardening enhanced by plastic deformation also might occur.

During experiments performed at 300 °C, several stress computations have been made from the curvature of mobile dislocations. The higher values, deduced from dislocation segments located near the pinning points, could be considered as representative of the unpinning stress. The lower values (20 and 28 MPa), which correspond to the instability threshold of two dislocation loops, would be more representative of the yield stress of the matrix; taking $\sigma \simeq 2\tau$ they are in reasonable agreement with the flow stress values of a 2024 alloy at 300 °C.

Experiments performed at 300 and 400 °C have also shown that the plastic behaviour of the matrix was very different at 200 °C and at these higher temperatures. At the latter ones, alloy elements are easily mobile and dislocation glide occurs at low stresses. Flow stress variation being weak, we can readily obtain reverse dislocation glide. Now, the question

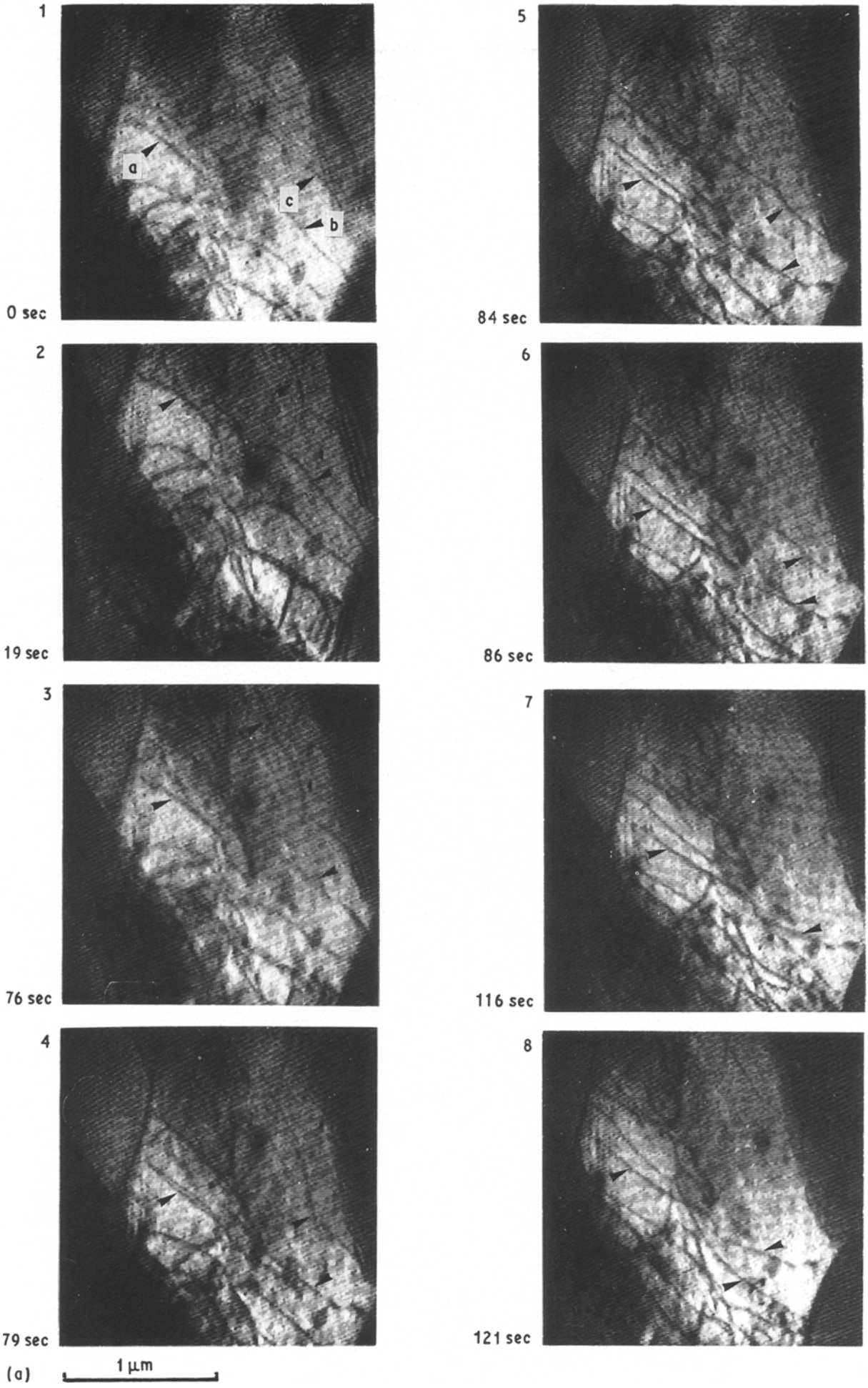
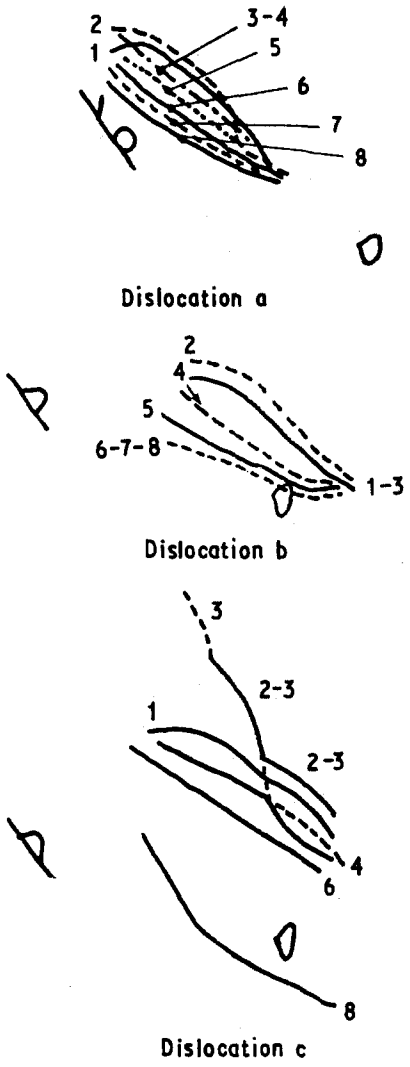


Figure 4 (a) A sequence of *in situ* electron micrographs during thermal cycling between 150 and 400 °C. (b) Schematic representation of successive positions of dislocations a, b, and c.



(b)

Figure 4 Continued.

arises whether thermal treatments usually performed on aluminium alloys significantly influence the mechanical behaviour of these MMC in the 300 to 400 °C temperature range. Further experiments are in progress to study this problem.

Acknowledgements

The authors thank J. Sevely, L.O.E.-C.N.R.S., Toulouse, for supplying MMC samples and valuable discussion on these materials. Louis Bernard is also thanked for difficult and careful sample preparation.

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Received 2 May
and accepted 29 September 1989