IN SITU TEM STRAINING EXPERIMENTS ON INCOLOY MA956 AT ROOM TEMPERATURE AND AT HIGH TEMPERATURES

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A direct experimental proof of a dislocation motion during plastic deformation of ferritic oxide dispersion strengthened (ODS) alloys is first given for ferritic alloy INCOLOY MA956. *In situ* straining experiments were performed at room temperature and at high temperatures (up to 1010°C) by means of high voltage electron microscopy (HVEM). Moving dislocations revealed a Burgers vector of a/2<111> type. Deformation at room temperature proceeds by jerky motion of dislocations on {110} slip planes. At high temperatures the dislocations show a completely different behaviour. A strong pinning of dislocations at dispersoids was observed, but the density of interaction points was low. Slip takes place on {112} and {123} planes. No subgrain formation was detected.

1. INTRODUCTION

Oxide dispersion strengthened (ODS) alloys have been developed for high temperature applications, e.g. energy power systems and gas turbine components [1]. Their design involves the understanding of the deformation processes under extreme service conditions, i.e. at temperatures above 1000°C. Modern theories of creep of dispersion strengthened alloys discuss the phenomenon of „departure side pinning" of dislocations at dispersoids, which can explain quite well the steady state creep behaviour of ODS alloys [2-5].

In the present paper we show the first *in situ* straining experiments on the ferritic ODS alloy INCOLOY MA956 within a wide range of temperatures. Direct observations of dislocation motion under stress improve the understanding of the deformation behaviour of ferritic ODS alloys.

2. EXPERIMENTAL DETAILS

A commercial ferritic ODS alloy INCOLOY MA956 with a chemical composition of (wt %): Fe - 19,89 Cr - 4.58 Al - 0.38 Ti - 0.51 Y_2O_3 was used for the investigations.

The alloy was supplied by Inco Alloys International as a hot extruded and recrystallized (1330°C/1h) bar, 20mm in diameter.

The very small tensile specimens (8×2×1mm) were cut parallel to the extrusion direction of the bar by spark erosion. On each specimen two holes were drilled for fixing the specimens to the grips. Specimens were ground to a thickness below 0.1mm, then one side was dimpled and finally double-jet polished using 10% perchloric acid in acetic acid (temperature of 11°C, voltage of 50V).

In situ experiments were performed at room temperature, 640, 700, 790, 880, 970 and 1010°C using the double-tilting stage [6] in the JEM-1000 high voltage electron microscope (1MV). Tensile load was applied in small increments. Moving dislocations were recorded on photographic film, as well as using the video facility. The results obtained are based on six successful *in situ* straining experiments.

3. RESULTS

3.1. As-received material

The INCOLOY MA956 bar exhibits a coarse elongated macrostructure with a grain aspect ratio up to 100:1 and a transverse grain dimension of about 3 mm. Conventional transmission electron microscopy revealed a fairly homogeneous distribution of yttrium-aluminium oxide particles (mainly $YAlO_3$) with a mean diameter of about 24 nm, a mean interparticle distance of about 150 nm and a volume fraction of 1.4% in a ferritic matrix. The dislocation structure was characterized by single dislocations pinned at dispersoids and mean dislocation density below $1\cdot10^{13}$ m^{-2} [7].

For a specimen thickness up to 1 μm the large areas transparent for electrons were observed, however no grain boundaries were detected. The crystallographic orientation of "quasi-single crystal" specimens in tensile direction was different for each specimen.

3.2. Room temperature *in situ* straining experiments

A typical image of the dislocation structure formed during *in situ* deformation of INCOLOY MA956 at room temperature is presented in Fig. 1a.

The slip process was documented on video tape of dislocations in motion. It shows that deformation proceeds by a two-step motion of dislocations with a Burgers vector of $a/2\langle 1\ \bar{1}\ 1\rangle$. The process is shown schematically in Fig. 1b. The activated slip systems were determined from the slip trace directions left by moving dislocations on a thin foil surface, taking into account their width and angles relative to the direction of the acting force. The main slip system activated at room temperature was $(110)[1\ \bar{1}\ 1]$. The glide dislocations formed slip bands in (110) slip plane. In regions with high dislocation density, a cross slip from (110) to (011) plane was observed, resulting from the high internal stresses inside the slip band. Many loops are visible within the slip band. As the (011) slip plane has a low orientation factor, screw dislocations do not move very far and revealed a typical jerky slip. One athermally activated step take place over distances greater than the obstacle distance, which is in agreement with athermal nature of the Orowan process.

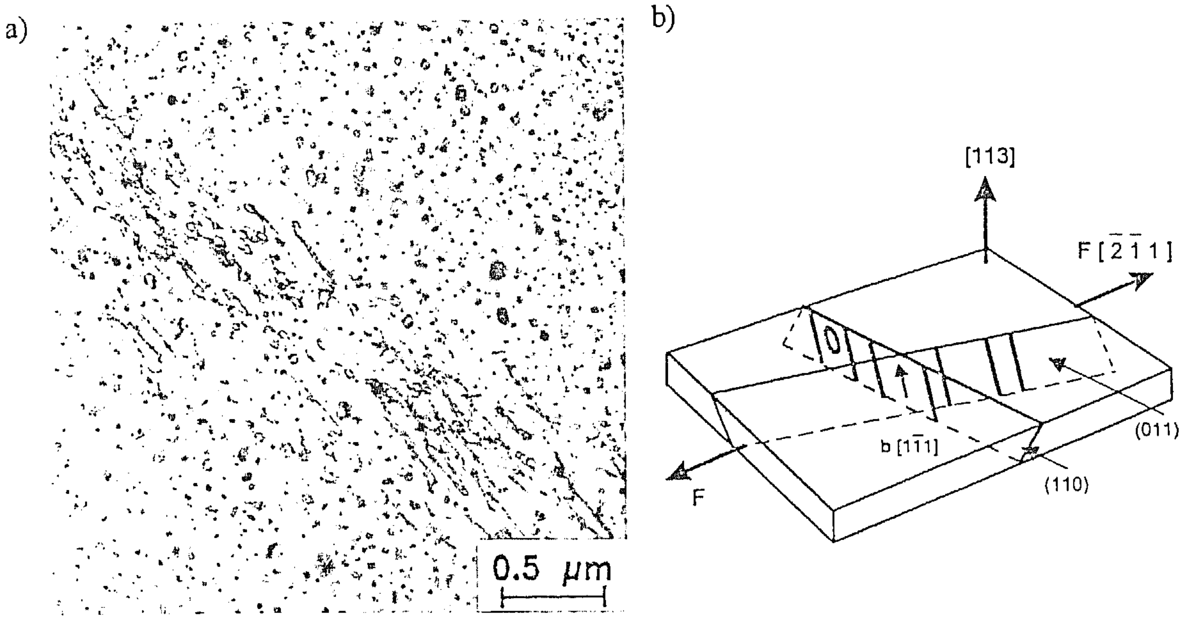

Fig. 1 : Specimen during *in situ* deformation at room temperature; a) typical dislocation structure, b) a scheme of deformation mechanism. Tensile axis $< 2\ 1\ \bar{1} >$.

3.3. High temperature *in situ* straining experiments

In situ experiments performed at temperatures above 640°C showed a completely different behaviour of dislocations (Fig. 2a). Dislocations exhibit a smooth curvature and move in a viscous manner. A detailed analysis of dislocation geometry in the point of interaction with dispersoids was restricted by a contrast of oxides and a larger dislocation width at high temperatures.

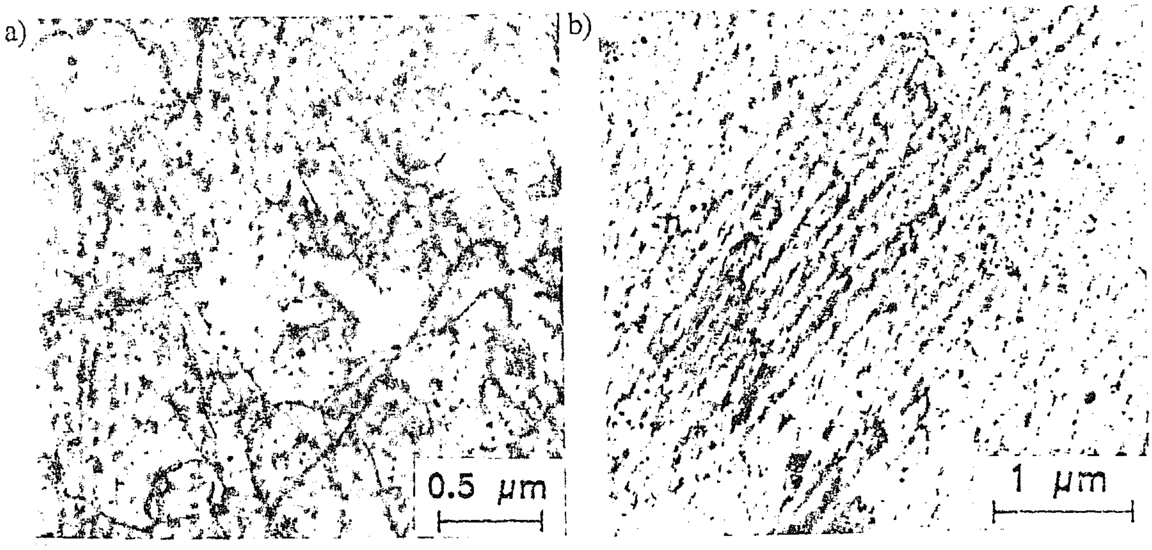

Fig. 2 : INCOLOY MA956 specimens during *in situ* deformation at high temperatures;
a) a dislocation structure at 790°C;
b) moving dislocations at a temperature of 970°C.

With increasing temperature, $\{1\ 1\ \bar{2}\}$ and $\{1\ 2\ \bar{3}\}$ slip planes become preferred. At a temperature of 790°C, the $\{1\ 1\ \bar{2}\}<111>$ slip systems with a maximum Schmid factor of 0.471 were dominant. Slip traces of dislocations observed at 970°C (Fig.2b) were in the direction <111>, parallel to their Burgers vector. Such behaviour can be a result of many preferred slip planes possible. However, a careful analysis showed that mainly $\{1\ 2\ \bar{3}\}$ <111> slip systems had a Schmid factor different from zero at that temperature. An experiment at 700°C revealed that rapidly unloading the specimen causes that the dislocations move back. The large distance the dislocation can go back after unloading suggests the existence of a strong internal elastic stresses.

The high temperature *in situ* experiments show that an obstacle distance is many times larger than that calculated from size and density of the dispersoids. In the „thermaly activated detachment" model [3] dislocations should jump very quickly to the next particle and than wait for detachment. Both is not found. The dislocations move viscously over several interparticle distances, and the waiting and travelling time are of the same order of magnitude. The viscous motion of the dislocations at high temperatures hints at the action of diffusion-controlled processes, which govern the dislocation mobility.

4. CONCLUSIONS

The described *in situ* experiments on INCOLOY MA956 revealed that there are strong differences in the dynamic behaviour of dislocations at room temperature and at high temperatures. A number of conventional transmission electron microscopy studies show that in creep deformed specimens dislocations are pinned at the departure side of the dispersoids. The quantitative microstructural data derived from such analyses should be taken very carefully, as the microstructural features controlling deformation at high temperatures are not always preserved down to room temperature.

The study shows that *in situ* straining in a high voltage electron microscope (HVEM) can be a powerful method for direct imaging of deformation processes at high temperatures.

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