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Obtaining and Observation of Bent Sub-Boundaries in the TiO_2 Rutile

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Abstract: This research concerns the study of mechanical behaviour of this material at high temperature. It is a question of observing dislocations microstructure in monocrystaline rutile by Tranmission Electronic Microscope (T.E.M.). This investigation method requires very thin foils, cut out in deformed sample. Thus it is necessary to develop a method to obtain an important dislocation density in restricted zone of the sample. The solution is to obtain three points bent sub-boundaries. For this purpose the chosen solution consists to stir only one slip system and then proceed to a polygonization.The slip system to stir is of the type {101}<î 01>. From a rutile monocrystal, samples have been cut, suitably oriented using X rays by laue method. After mechanical polishing, samples have ben strained in three points bent at 1000°C then annealed at 1300°C during 12 h. The sub-boundaries were observed by optical microscope by eth-pits which consists to chemical polishing by alkaline fusion in KOH followed by an immersion in boiling sulphuric acid during 2 to3 min. It is sub-boundaries of very weak disorientation. Practically all concentrates in central zone of the sample. The rutile being a ceramic chemically weak stired, thin foils have been obtained by ionic bombardment thinning method. The T.E.M. observation have allowed to characterize the dislocations forming the sub-boundaries corresponding to dislocations arrangement of corner type, located in the same plan.

Key words: Microstructure, T.E.M., Polygonization, alkaline fusion, sulphuric acid

INTRODUCTION

The oxide of titanium TiO_2 (rutile) is regarded at the present time as one of the major element of ceramic ranges. This research is focused on the determination of this material mechanical behavior at high temperature.

The aim of this research is to obtain bent sub-boundaries in rutile monocrystal in order to observe their microstructure under the Transmission Electron Microscope (T.E.M.).

For this purpose, the solution which consists to activate only one slip system and then proceeding to a polygonization is chosen.

The rutile presents two principal slips systems (Blanchin *et al*., 2002):

- The system {101}<î 01>: This system is easily activable starting from 875°K.
- The system {110}<001>: This system is activated starting from 900°K but for a strain much higher than the previous.

The crystallographic orientation of samples to be deformed, has to be judicious in order to activate the only slip system of {101}< î 01> type.

MATERIALS AND METHODS

Preparation of the samples to be deformed: A rutile monocrystal, in the shape of cylinder elaborated by Verneuil method,was used.The growth axis which is also the cylinder axis is the (100) direction. The cutting of samples intended for bent deformation is carried out using the set with diamonds grinding stone saw.The crystallographic orientation of the samples is carried out by the diagrams of x-rays diffraction using Laue method. To cut the test-samples, one fixes the monocrystal starting block on an adaptable goniometric assembly at the same time on the installation of x-rays diffraction and on the saw. This assembly enables us to direct the monocrystal block and to cut sections in a chosen crystallographic orientation. In this study the samples of deformation whose laterales faces have a determined crystallographic orientation, will be cut. From initial crystal, samples of parallelepipedic form (L.l.e) were obtained. Their crystallographic orientation is represented on (Fig. 1).

This orientation gives a maximum Schmid factor for the slip system {101}< î 01>.

The strain knives of bent machine are placed on the two parallel faces of (001) type.The axis of the constrain is then according to <100> direction.

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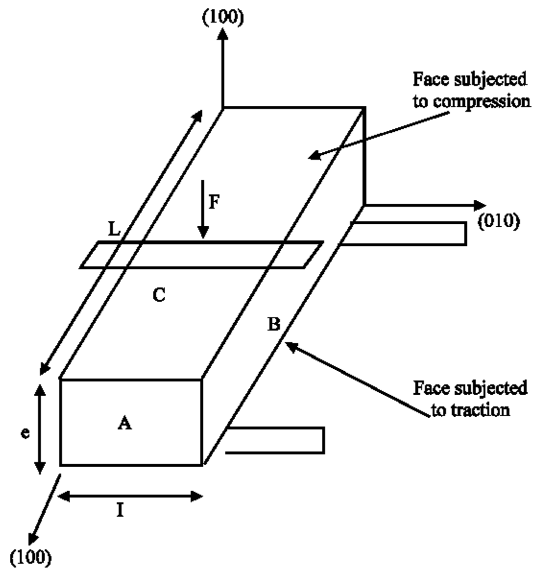

Fig. 1: Sample of deformation

In bent deformation the two faces of the sample to be deformed, in contact with the knives, are subjected either to a tensile strain or to a compression strain. A starter of crack on the face subjected to traction can be the cause of sample rupture. Whereas on the other parallel face, the compression strain tends to close again the cracks.

The higher C face will be mechanically polished by a reference treatment which consists to polish by paper carborandum: grain 320, then with the carborandum: grain 500, then with the carborandun: grain 1000, then with the diamond paste: 15 μm and finally with the diamond paste: 7 μm.

The lower C face, in addition to the reference treatment, will be finished with diamond paste 1 μm (face subjected to traction).

The two B faces will be polished by a reference treatment, whereas the two A faces remain rough of cut.

Three points bent deformation: The deformation machine used is Instron type 1195 with a capacity of 100 kN, equipped with a Pyrox furnace which can reach a temperature of 1600°C.

- The sensors have a sensitivity of 5 mV μm^{-1}.
- The deformation speed used is of 0,05 mm mn^{-1}.

The rutile has a plastic deformation for a temperature higher than 875°C. In the case study, we deformed at a temperature of 1000°C and under air. The speed of rising temperature being 200°C/h.

Annealed polygonization: After deformation, samples have subjected to an annealing of 12 h at a temperature of 1300°C. This annealing should allow a rearrangement of the dislocations introduced by the plastic deformation, in sub-boundaries.

Corrosion figures: Obtaining the eth-pits consists of a selective attack of the deformed sample surface to be able to observe emergences of dislocations, under the optical microscope.

The experimental method is as follow:

- Chemical polishing by alkaline fusion in potash KOH: the Potash is made completely anhydrous by holding the temperature of 460°C during approximately 4 h. A short stop of the heating allows the solidification of the bath. The sample is then put on the surface of the bath and carries the unit to 460°C during approximatively half an hour. Then take out the sample avoiding the thermal shocks. The residual film on the sample surface is dissolved by immersion in distilled water. This alkaline fusion is carried out in an alumina container.
- Obtaining the eth-pits by immersion in the ebullient sulphuric acid during 2 to 3 min.

T.E.M. observation: The choice of thinning method for T.E.M. observation was guided by the fact that the rutile is a ceramic chemically weak stired. Thus thinning method by argon ionic bombardment was chosen. This preparation of thin foils is carried out in the following way (Blanchin and Fisant, 1999) using the optical microscope, one locates the sample zone containing sub-boundaries (zone of approximately 2 mm thickness). Cuting this zone using a wire saw with circulation of abrasive (boron carburizes). The obtained foil is then polished mechanically until a thickness of about 50 to 60 μm, measured with the micrometer caliper. To carry out this operation, the foil is held by joining on a glass plate thanks to a special synthetic cement. The cement is then dissolved with alcohol. The foil is then assembled on the slide of an ionic thining with double beam. This thinning enables us to obtain areas of about 1000 Angström thickness, transparent with the electrons of 100 kev energy.

RESULTS AND DISCUSSION

Deformation in three points bent: To have an appreciable density of dislocations without a risque of break, we stopped the deformation at the beginning of the plastic range. thus a curve, giving the force F applied according to the displacement of the higher knife i.e., according to the arrow f, was obtained. This curve present an elastic zone and a plastic zone.

Observation of arrangements of dislocation by eched figures: The optical microscope observation show on the two faces (010) the emergences points of the dislocations lines in the form of rhombus shape, arranged on sub-boundaries of very weak disorientation. These sub-boundaries are all concentrated in the center of the sample, over a width of approximately two millimeters (Fig. 2-4).

Observation of the structure of a sub-boundaries with the T.E.M.: The sub-boundaries being concentrated in the central part of the sample, the thin foil was prepared starting from this zone. The T.E.M. sub-boundaries observation, enabled us to note that they are made of an alignment of parallel corner type dislocations, located in the same plan.

The following micrography (Fig. 5), presents the microstructure of a sub-boundary, the plan of the thin foil being close to (100).

Fig. 2: Bent sub-boundaries

Fig. 3: Bent sub-boundaries

Fig. 4: Bent sub-boundaries

Fig. 5: Mcrostructure of sub-boundary

CONCLUSION

The aim of this study was to observe with the T.E.M. the structure of bent sub-boundary in monocrystal of rutile. For that, we initially deformed in three points bent, test-samples of rutile judiciously directed in order to activate only one slip system and then proceeded to a polygonization. Sub-boundaries of very weak disorientation, concentrated in a mean central part of the sample, was obtained. Observation of a sub-boundary by the T.E.M., showed that it is about an alignment of corner type dislocations, located in the same plan.

REFERENCES

Blanchin, M.G., P. Faisant and C. Picard, 2002. Phys. State Sol., 60: 1.

Blanchin, M.G. and P. Faisant, 1999. Revue Phys. Applied, 14: 619.