

Dynamic Study of the Thermoelastic Transformation of Cu-Zn-Al single crystals by X-Ray diffraction

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Abstract :The martensitic transformation of Cu-Zn-Al- single crystals has been followed in situ and in real time by synchrotron X-ray topography. We have shown that at the M_s temperature, the transformation proceeds by nucleation and growth of transformation variants associated by self-accomodating pairs. The nucleation of these variants is triggered by the crystal substructure. When the transformation is multivariant we have displayed the development of elastic stresses, accompanying the growth of variants, inducing a reversible curvature of the lattice planes.

1. INTRODUCTION

The synchrotron X-ray topography is an experimental method which can be successfully applied to the study of martensitic transformation. In spite of its resolution limited to some microns, this method is the only one which brings at the same time information on the crystallography, on the long range stresses accompanying the transformation and on the defect structure of the crystals. The white nature of the synchrotron beam allows to register simultaneously, on a same film, the diffraction diagrams of the two phases of a crystal submitted to thermal cycling and its low divergence allows to display and to characterize the crystal defects with their mutual interactions. The high flux of the synchrotron radiation allows to study the phase transitions and the structure evolutions of the crystals "in situ and in real time". These specific properties make this method complementary to the optical and electron microscopic methods whose typical results can be found in references [1-5].

The thermoelastic martensitic transformation of Cu-Zn-Al single crystals has been followed by synchrotron X-ray topography. In this paper some characteristic evolutions of the crystal structure during the phase transformation are given. In particular the role of the defects of the parent crystal on the selection of the transformation variants as well as the development of elastic stresses accompanying the transformation will be emphasized.

2. EXPERIMENTAL TECHNIQUE

Detailed information on the techniques used in this study are available in reference 6.

Disk-shaped single crystals with a diameter of 10^{-2} m and a thickness of 10^{-4} m were cut from an ingot obtained by a modified Bridgman technique. They were mechanically and electropolished. Their M_s point is close to 263 K.

With the characteristics of the synchrotron white beam the time exposure to record diffracted beams on films is the order of 1 to 5 seconds.

Experiments were made in a Peltier cell designed in such a way that the X-ray beam can pass through. When the first nucleus is detected the temperature is slowly decreased in steps of 0.2 K. Topograms

(diffraction diagrams) are recorded at each step. The reverse transformation is studied similarly. In this paper the β and 9R terms are used to designate the high and low temperature phases.

3. RESULTS AND DISCUSSION

3.1. Nucleation and growth of the 9R low temperature phase

The in-situ study of Cu-Zn-Al crystals submitted to thermal cycling shows that generally boundaries and subboundaries of parent crystals are preferential nucleation centers. In this study we have focussed our attention to the role played by the subboundaries which are the prevalent crystal defects observed in single crystals.

Figure 1 shows the diffraction diagram given by a Cu-Zn-Al single crystal during the $\beta \rightarrow 9R$ transformation at 262.5 K, temperature slightly lower than the Martensitic start temperature ($M_S = 263$ K). The β and 9R phases diffract simultaneously. They are marked by an arrow.

The circular diffraction spots are given by the β parent phase. The white traces inside these spots correspond to the transformed parts of the single crystal which are in the 9R martensitic structure and which diffract in the form of elongated rectangular diffraction spots.

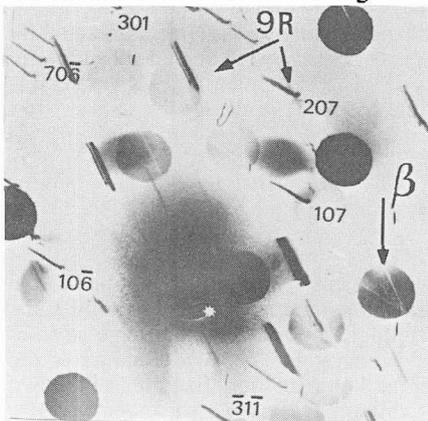


Figure 1

Diffraction diagram given by the crystal at 262.5 K

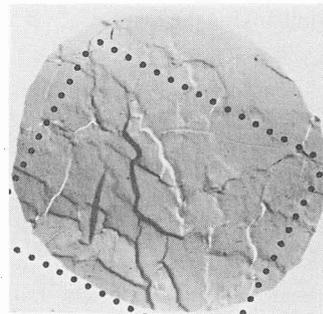


Figure 2

Photographic enlargement of the 231 β diffraction spot

The photographic enlargement of the 231 β diffraction spot is given in figure 2. Subboundaries are visible either in black or in white according to the misorientation sense of subgrains. The β crystal structure evolution during the $\beta \rightarrow 9R$ transformation is given in figure 3. Four stages are presented in Figures 3a to 3d which are the photographic enlargements of the framed part of the 231 β diffraction spot visible in figure 2.

The first variant appearing during the $\beta \rightarrow 9R$ transformation of the single crystal is the variant called 1. At 262.5 K this transformation variant 1 induces the nucleation of the variant 2 figure 3b. During its growth this last variant 2 induces the nucleation of a new variant 1 figure 3c which, in its turn, induces the nucleation of two new variants 2 figure 3d. All the nucleation centers activated have been marked in figure 3a by a black dot. An arrow shows that they are located on subboundaries.

The determination of the crystallographic orientations of the transformation variants shows that they have not a random orientation among the 24 orientations possible deduced from the crystallographic orientation of the parent crystal [7]. We have shown [6] that the 1-2 pair of martensitic plates, formed during the transformation, is a self-accommodating pair belonging to the same self-accommodating group.

The dynamic study shows that the $\beta \rightarrow 9R$ transformation progresses by nucleation and growth of variants associated by self-accommodating pairs. The nucleation centers, generally situated on crystal defects, are activated when they come into contact with a transformation variant (subboundaries in this study).

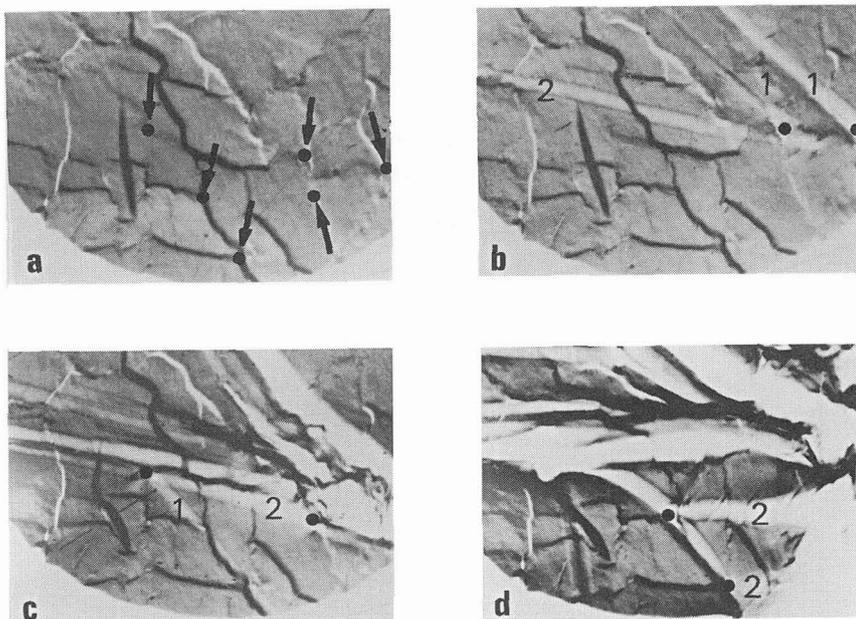


Figure 3
 3a : initial state - 3b : 262.5 K - 3c : 262.2 K - 3d : 261.5 K

3.2. Long range stresses due to the $\beta \rightarrow 9R$ transformation

To give a description of the crystal structure evolution during the $\beta \rightarrow 9R$ transformation, three photographic enlargements of a diffraction spot, characteristic of the evolution of the crystal structure, have been grouped in figure 4. Figure 4a shows the general aspect of the crystal structure near the M_s point, when the two phases are present. The circular β $23\bar{1}$ diffraction spot in which the position of the variants 1-2 is clearly distinguished and the diffraction spots given by these two variants indicate that the crystal structure (β and $9R$) is comparable to the crystal structure before transformation. (Figure 2). When the transformation progresses the growth of the self-accomodating variants involves elastic stresses which deform the crystalline lattice of the two phases. These stresses are displayed by the distortion of the $23\bar{1}$ diffraction spot. (Figure 4c). In figure 4b the diffraction spots of the β and $9R$ phases are close. Undulations of the $9R$ diffraction spots reflect the lattice rotations of the $9R$ phase. These rotations have been estimated and they can reach the degree. These stresses are characteristic of a multivariant transformation even if the variants are self-accomodating variants. When the transformation is monovariant as it is the case during the training by tension, for example, these curvatures of the lattice planes are not observed. In Figure 5 we show the diffraction spots of a Cu-Zn-Al single crystal submitted to a training treatment. Figure 5a is the β diffraction spot before transformation, 5b and 5c are respectively the β and $9R$ diffraction spots during the transformation at 260K.

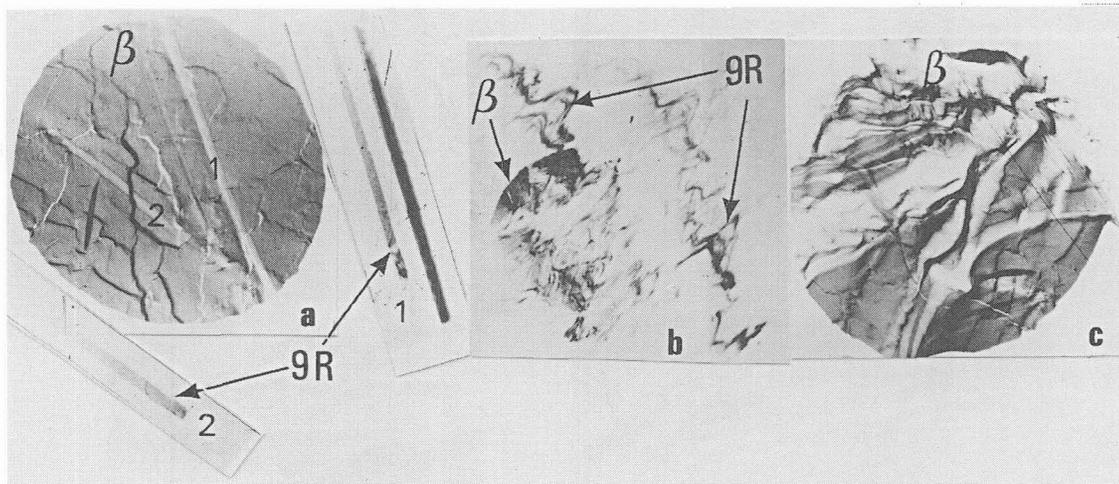


Figure 4

Elastic stresses appearing during the martensitic transformation .
 4a : at the M_s temperature ; 4b and 4c : during the transformation. The elastic stresses which deform the lattice of the two phases are clearly displayed by the topograms.

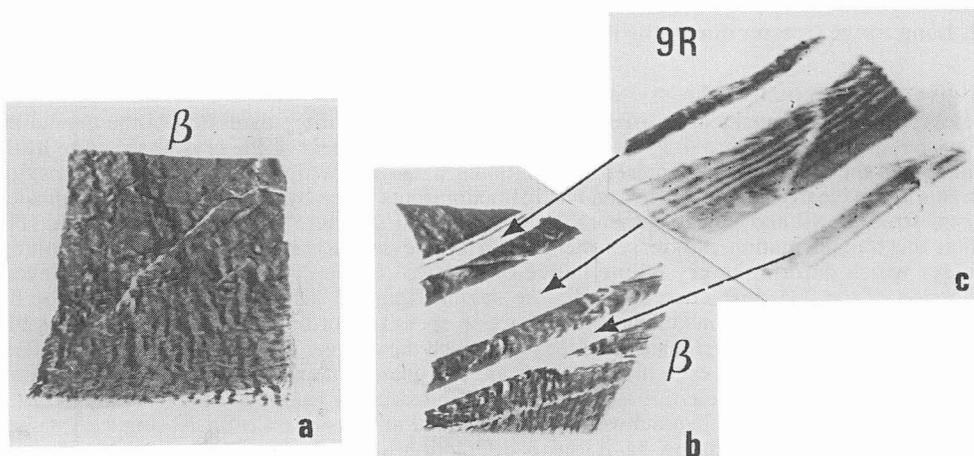


Figure 5

Single crystal submitted to a training treatment. 4a : β diffraction spot before transformation ; 4b and 4c : β and 9R diffraction spots at 260 K during the $\beta \rightarrow 9R$ transformation

4. CONCLUSIONS

The martensitic transformation of large single crystals of Cu-Zn-Al alloy has been studied in situ and in real time by synchrotron X-ray topography. We have displayed two new experimental results.

Near the M_s point, the β to 9R phase transformation progresses by nucleation and growth of self-accommodating variants associated by pairs. The nucleation centers are generally situated on crystal subboundaries they are "activated" when a transformation variant meets the subboundary.

The development of elastic stresses resulting from the growth of the transformation variants in a multivariant transformation has been displayed. Their persistence after total transformation in 9R phase can contribute to the backward movement of the interphase boundaries in the reverse 9R $\rightarrow\beta$ transformation.

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