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ON THE FORMATION OF TWINNED PRECIPITATES IN Al-Ge ALLOYS

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Résumé - Une meilleure compréhension de la précipitation de Ge dans Al a été obtenue par une utilisation systématique de la microscopie en haute résolution. La formation des aiguilles maclées qu'on observe le long de $\langle 100 \rangle_{Al}$ et $\langle 110 \rangle_{Al}$ peut être reliée à l'existence de quelques configurations atomiques particulières. On montre aussi que le maclage sert à réduire le nombre de lacunes utilisées lors de la nucléation des précipités.

Abstract - Atomic resolution microscopy has been used systematically to obtain a better understanding of Ge precipitation in Al. The formation of the observed twinned $\langle 100 \rangle_{Al}$ and $\langle 110 \rangle_{Al}$ needles can be related to the existence of a few special configurations. It is shown that twinning serves during nucleation to reduce the need of vacancies.

1 - INTRODUCTION

A solid state precipitation reaction is a convenient method for forming heterophase interfaces in materials. Since the precipitating phase nucleates and grows in the bulk of a supersaturated solid solution a clean contamination-free condition for the interface formation is assured.

Aluminum-germanium is a particularly interesting system for study because many different orientation precipitate morphologies develop and several different orientation relationships are found. It also provides metal/semi-conductor interfaces. Some of the factors important in determining the shapes, orientation relationships, internal and interface structures can be deduced from high resolution studies of the structure of needle, rod and lath precipitates observed end-on.

2 - EXPERIMENTAL

After ageing for a week at 550°C, 3 mm discs of an alloy of nominal composition Al - 1.1% at. Ge were quenched to -60°C in order to create vacancy supersaturation. Precipitation was ensured either by bulk or in-situ ageing for 4 h between 220°C and 260°C. High resolution imaging of precipitate cross-sections was performed using the Berkeley Jeol ARM 1000 operating at 800 kV.

3 - RESULTS

The overall precipitate morphology and distribution in quench/aged Al-Ge is shown in Fig.1. Needles can be found either parallel to the $\langle 100 \rangle_{Al}$ or $\langle 110 \rangle_{Al}$ axis. The influence of vacancy supersaturation on the type of precipitate nucleated has been directly examined in thin foil experiment [1]. It has been observed that in thin regions of a foil where substantial loss of vacancies occurs, needles are the predominant morphology nucleated. Among all the precipitate configurations, multiply-twinned precipitates (MTP) is the most frequent one.

Fig. 1 : A typical micrograph from a quench/aged Al - 1.1% Ge alloy shows long needle precipitates lying along $\langle 100 \rangle_{\text{Al}}$ directions.

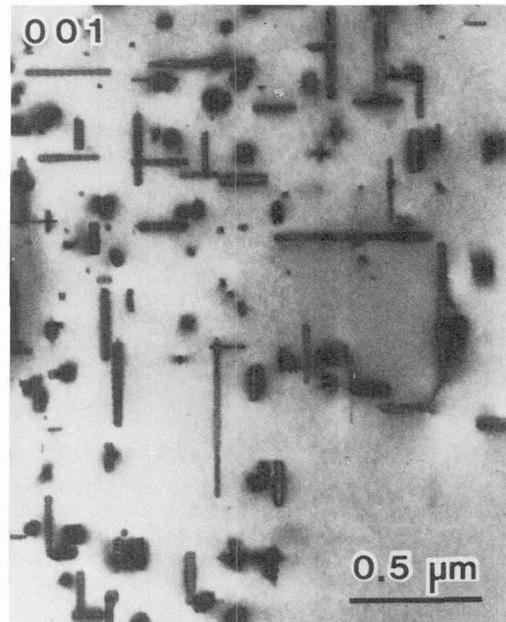


Figure 2 shows a high resolution micrograph of a typical example of such a MTP. Such images simultaneously give information on the shape, substructure, orientation relationship and interface structure of precipitates. This Ge particle exhibits one set of parallel twins and an interface with the Al matrix that is faceted on $\{111\}_{\text{Ge}}/\{100\}_{\text{Al}}$ and $\{111\}_{\text{Ge}}/\{310\}_{\text{Al}}$ planes.

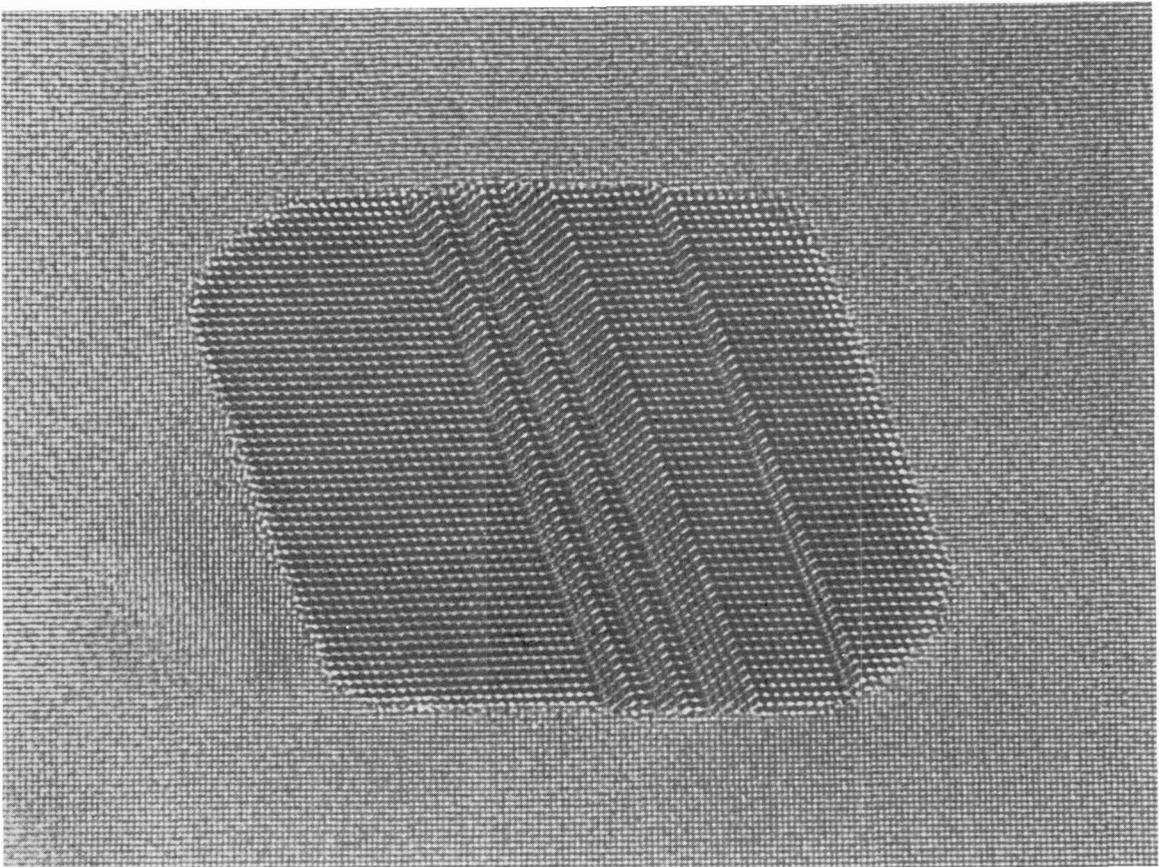


Fig. 2 : High resolution micrograph in which a needle is observed in cross-section. The axis of the needle is $\langle 110 \rangle_{\text{Ge}}$ parallel to a $\langle 100 \rangle_{\text{Al}}$ direction. No relaxation into a dislocation structure is apparent at either type of interface.

As already reported /1/, the faceting of the precipitate occurs mostly on $\{111\}_{\text{Ge}}$ planes and usually exhibits contrasts consistent with twinning or a change in stacking sequence at the interface between aluminum and germanium.

When twinning occurs in different planes sharing a common $\langle 110 \rangle$ direction, five internal twin-related segments are found. Figure 3 (a) shows a high resolution micrograph of a cross-section of a multiply-twinned precipitate found in a bulk-aged sample. The axis of the needle is $\langle 110 \rangle_{\text{Ge}}$ parallel to a $\langle 100 \rangle_{\text{Al}}$ direction. Since the five twins converge exactly on the same point, the intersection area has a special crystallography : a model of this configuration is shown in Fig. 3 (c) and the corresponding simulation in Fig. 3 (d). The twins meet in a common center imaged as a five-member ring of (black) Ge atom pairs. This kind of ring has already been observed in materials with diamond or face centered cubic structure, e.g. /2,3/.

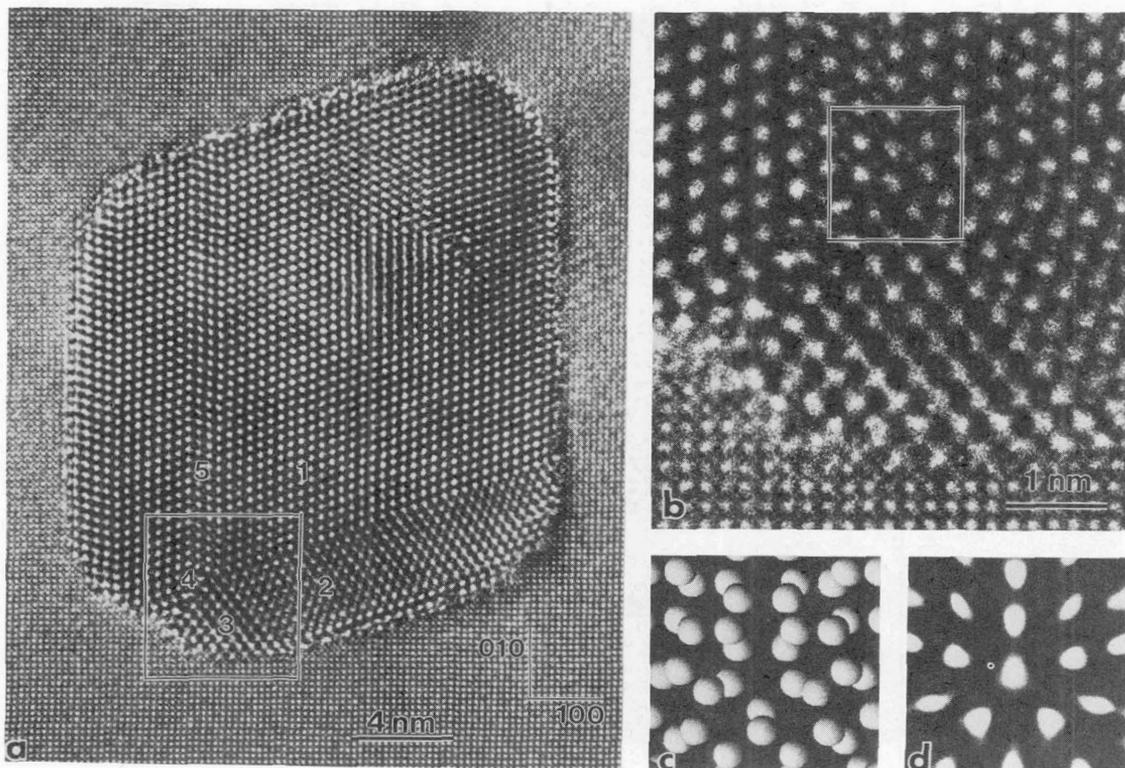


Fig. 3 : Bright field micrograph showing the morphology of Ge precipitate in a bulk-aged sample. Note the alignment along the $\langle 100 \rangle$ direction in Al (for the simulation (d), defocus = - 80 nm, thickness = 5 nm).

Due to the obligation to accommodate the 7.5° gap left by five twin segments enclosing an angle of 70.5° each (352.5°), pentagonally-twinned particles are usually found to be asymmetrical. However it is possible to find regular particle with a pentagonal symmetry (fig. 4).

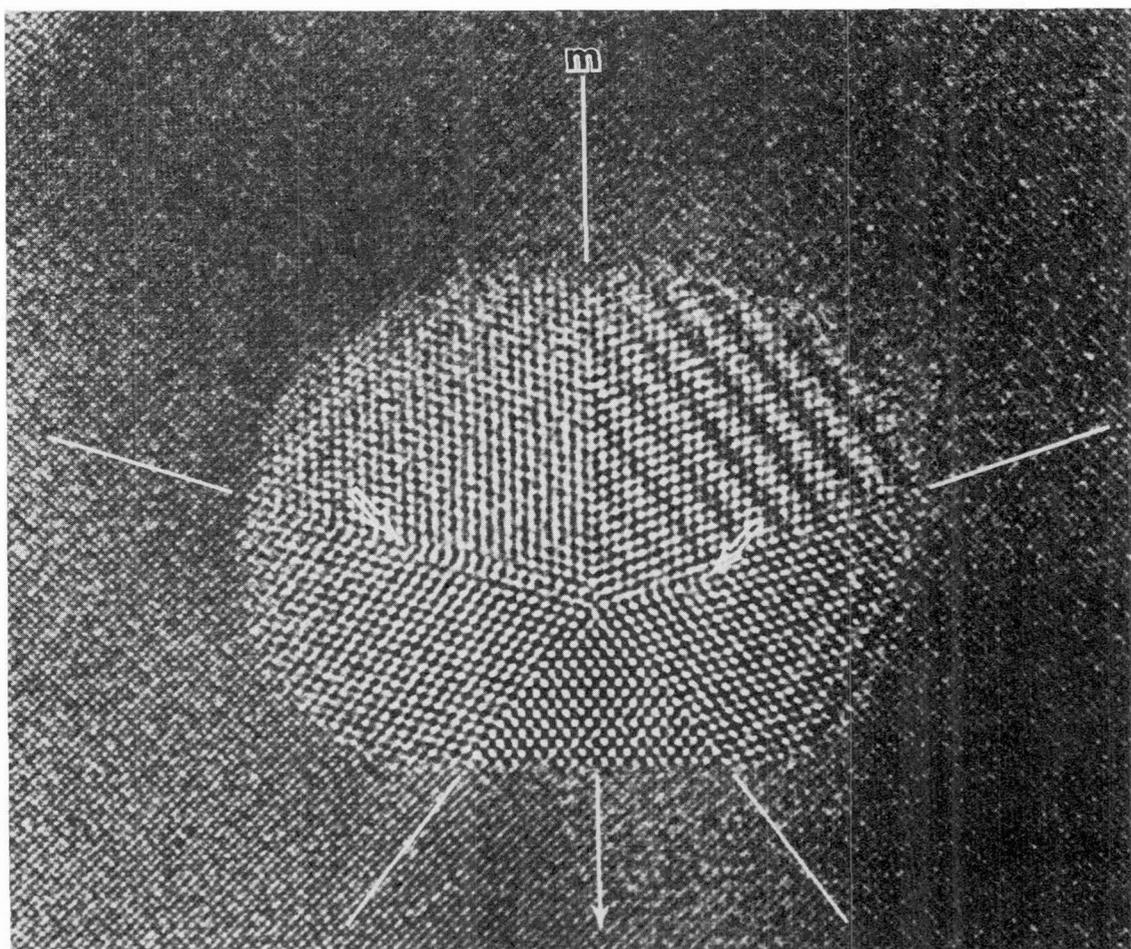
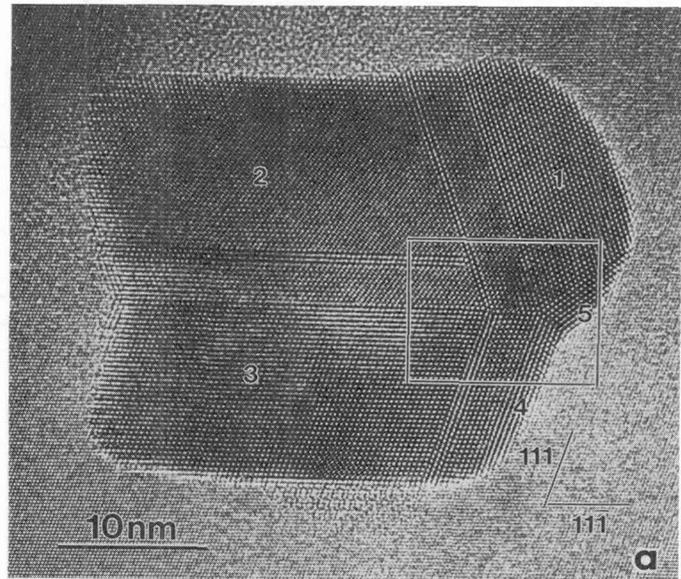
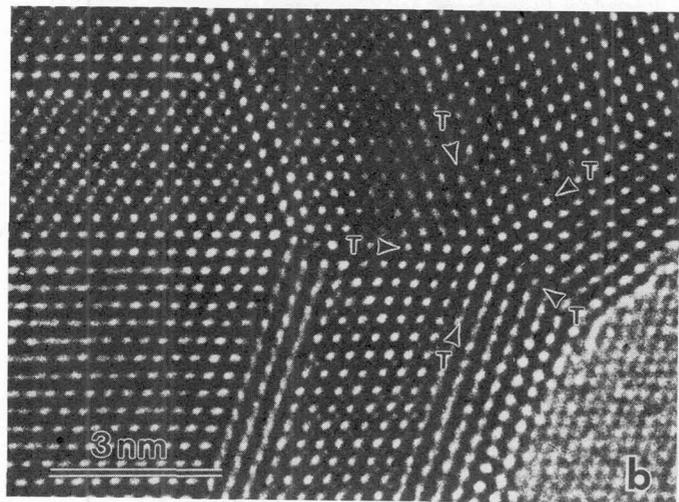


Fig. 4 : Ge precipitate with regular arrangement of five twin segments meeting at a common center. The vertical mirror plane indicated by a line (m) is shared by the Al matrix, the triangular twin segment at the bottom of the Ge particle and the entire particle morphology, including the extra half planes inserted at the inclined twin boundaries to relieve the 7.5° gap. Precipitates with such a high degree of perfection in the arrangement of internal defects are unusual.

Fig. 5 : High resolution micrograph of a $\langle 110 \rangle_{Al}$ needle with the so-called "mushroom" configuration. The head part appears to have a pseudo five-fold symmetry.



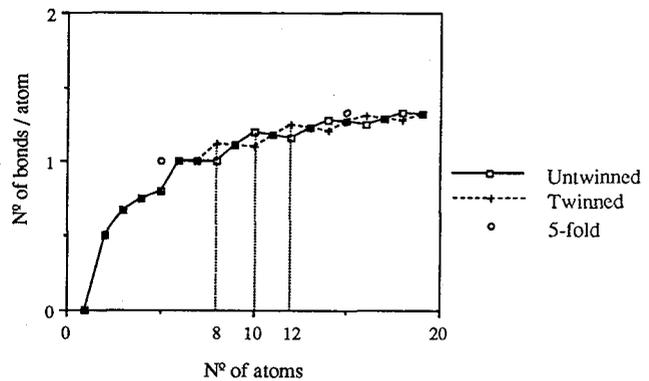
In an analogous manner to $\langle 100 \rangle_{Al}$ needles, $\langle 110 \rangle_{Al}$ needles also display multiply-twinned morphology. Figure 5 is an example of such a precipitate. In the enlargement in fig.5 (b), the convergence of the five twin segments is clearly visible.



4 - VACANCIES AND TWINNING IN NUCLEATION

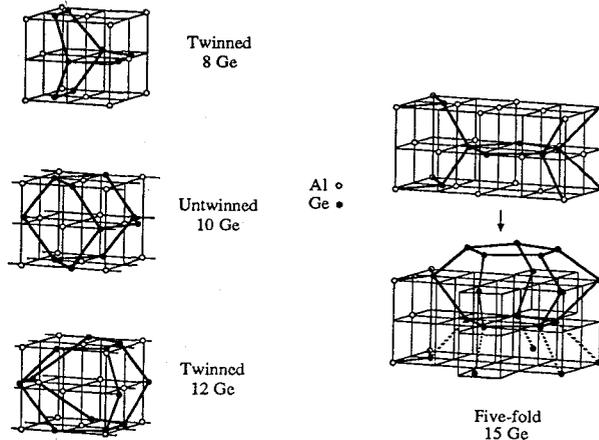
In order to accommodate the large disparity in the atomic volumes of aluminum and germanium (16.6 \AA^3 and 22.6 \AA^3 respectively), precipitation and growth need an excess of vacancies, usually introduced by quenching, e.g. /4/. The ease of nucleation will be then proportional to the concentration of vacancies or at the least the need a particular configuration has for vacancies. Thus it is postulated that nucleation occurs by a process which tends to reduce the number of vacancies used by increasing the number of Ge-Ge bonds per atom. It is possible to construct systematically the possible configurations leading to maximum number of bonds for a given number of atoms in the diamond-cubic structure. As shown in figure 6, when the number of atoms involved is 8, 10 or 12, special closed configurations, or cages, appear which have a higher density of the precipitate nucleus. The first complete nucleus with maximum density is the eight-atoms cage. It consists mainly of six-membered rings with the so-called boat configuration and its extension will lead to a twinned crystal. This is not the case for the second cage in which ten

Fig. 6 : A plot of the maximum number of bonds possible for a given number of atoms in the diamond-cubic structure is followed by the different atomic configurations corresponding to points on the curve. For some of these, twinning leads to an increased number of bonds per-atom and thus a reduced need for vacancies.



atoms forming six-membered rings constitute the chair configuration known to give rise to an untwinned crystal. The third configuration with twelve atoms is in fact the continuation of the first configuration with eight atoms, as the fourth is the next step after the ten-atoms cages, and so on. As indicated on fig. 6, the cage resulting from a five-atom ring is only completed for fifteen atoms and doesn't appear to be directly at the origin of particle with five-fold symmetry.

From this considerations, it is clear that twinning serves during nucleation to maximize the number of Ge-Ge bonds and thus to reduce the need of vacancies, whereas during growth accidental twinning strongly influences the developing morphologies including the pentagonally twinned configuration.



5 - CONCLUSIONS

The present study shows that during precipitation of Ge in Al, one of the predominant factors for the formation of the nucleus is the demand for vacancies. The configuration which appears to follow this criterium best consists of only eight atoms and invariably leads to the formation of a twinned crystal. Further work is in progress to include rationalization of all observed morphologies in function of the different local vacancy concentrations and different sequences of twinning.

The results also show the convenience of solid state precipitation for study heterophase interfaces in materials.

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