TEM analysis of deformation induced dynamic nanocrystallization in an amorphous CoTi alloy

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Nano-sized crystals in an amorphous matrix are considered to change the mechanical properties of an amorphous alloy. Therefore, it is of special interest to manipulate and control both the size and structure of nanocrystals. In many cases, nanocrystals are formed by special heat treatments. Here, we show that nanocrystals can emerge out of the amorphous phase during severe plastic deformation. This can be revealed by studying the composition and the atomic structure of the crystals using different transmission electron microscopy (TEM) methods.

In our work pure components are used to make a Co$_3$Ti alloy. Homogenisation at 950°C for 100 hours leads to a L1$_2$ long range ordered single phase alloy. The samples were deformed by high pressure torsion (HPT) using 4 GPa pressure and 80 rotations. After deformation TEM imaging with a Philips CM200 yields both crystalline and amorphous regions present in the samples. The situation is similar to that of Zr3Al, a L12 alloy, that can be made amorphous by severe plastic deformation [1]. The striking result of the present study is that in the amorphous regions nanocrystals of about 2-20 nm in size are embedded. Their average size is about 12 ± 0.5 nm and they exhibit a volume fraction of about 2 ± 1% (cf. Fig.1). From the analysis of bright field (BF) images taken from different sample sections it can be concluded that the nanocrystals have a spherical shape.

The chemical composition of the nanocrystals is analysed in a FEI Titan microscope by electron energy loss spectroscopy. The Ti atomic concentration for individual nanocrystals is 18 % higher than those of the surrounding amorphous matrix. This indicates that the nanocrystals are of the Laves phase Co$_{2.1}$Ti$_{0.9}$. Therefore we conclude that the nanocrystals are not retained crystalline material but rather formed during deformation by dynamic crystallisation.

For structural information high resolution transmission electron (HRTEM) images of the nanocrystals are acquired. The HRTEM images show lattice planes according to the Kagome layers of Laves phases (cf. Fig.2). Nevertheless, the analysis of the stacking sequence of the Kagome layers A, B and C does not reveal unambiguously the corresponding Laves phase due to a high density of faults. The structure can be described either by a faulted Co$_3$Ti (stacking sequence ABC) or a faulted Co$_{2.1}$Ti$_{0.9}$ (stacking sequence ABAC). In order to have reference images of an unfaulted Laves phase the as-cast alloy containing the Co$_{2.1}$Ti$_{0.9}$ Laves phase (C36) was studied. Therefore HRTEM images are acquired with a CM30 microscope. By using the Kikuchi patterns the sample was tilted to a [100] pole of Co$_{2.1}$Ti$_{0.9}$. Fig.3 shows the corresponding HRTEM image of the Kagome layers with an unfaulted ABAC stacking order.


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Fig. 1: BF image of the amorphous phase of the Co3Ti alloy with embedded nanocrystals formed by dynamic crystallization. The nanocrystals show bright and dark contrast relative to the contrast of the amorphous phase.

Fig. 2: HRTEM image of a nanocrystal showing Kagome layers with frequently occurring stacking faults.

Fig. 3: HRTEM image of the Laves phase in the as-cast alloy showing the unfaulted ABAC stacking sequence of Co2.1Ti0.9.