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Isothermal martensitic and pressure-induced δ to α′ phase transformations in a Pu-Ga alloy

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A well-homogenized Pu–2 at.% Ga alloy can be retained in the metastable face-centered cubic δ phase at room temperature. Ultimately, this metastable δ phase will decompose via a eutectoid transformation to the thermodynamically stable monoclinic α phase and the intermetallic compound Pu_3Ga over a period of approximately 10,000 years [1]. In addition, these low solute-containing δ-phase Pu alloys are metastable with respect to an isothermal martensitic phase transformation to the α′ phase during low temperature excursions [2, 3] and are also metastable with respect to a δ → α′ phase transformation with increases in pressure [3-5]. The low temperature δ → α′ isothermal martensitic phase transformation in the Pu–2 at.% Ga alloy only goes to ~25% completion with the resultant ~20 μm long by 2 μm wide lath-shaped α′ particles dispersed within the δ matrix. In recently reported studies, Faure *et al.* [4] have observed a δ → γ → α′ pressure-induced phase transformation sequence during a diamond anvil cell investigation and, based on x-ray diffraction and density and compressibility experiments, Harbur [5] has concluded that both α′ and an amorphous phase are present in samples that were pressurized and recovered. In this work, a large volume moissanite anvil cell is constructed to permit the pressurization and recovery of specimens of a size suitable for TEM and electron diffraction studies. The cell, shown in Fig. 1, has an overall diameter of 101.6 mm, a moissanite anvil diameter of 9.00 mm, a culet size of 3 mm, and a spring steel gasket 0.5 mm thick with a hole diameter of 2.5 mm. A 2.3 mm diameter by 100 μm thick sample of δ-phase Pu–2 at.% Ga is compressed at a rate of approximately 0.05 GPa/minute to ~1 GPa to induce the phase transformation to α′. Optical microscopy of the recovered specimen reveals a very fine microstructure that appears to be single phase, although the resolution of this technique is insufficient to differentiate between single and multiple phases if the grain size is below approximately 1 μm. X-ray diffraction, using a laboratory Cu K_α source with wavelength of 1.542Å, shows the monoclinic reflections from the α′ phase, strong peaks from the aluminum specimen holder, and weak peaks from the face-centered cubic δ phase as shown in Fig. 2. The recovered specimen is prepared for TEM and electron diffraction studies as described in Moore *et al.* [6]. TEM reveals small regions of δ phase with a very high dislocation density interspersed between the 10 – 100's nm α′ grains as shown in Fig. 3. Electron diffraction, shown in the insert in Fig. 3, clearly reveals the presence of the δ phase. This microstructure is in contrast to the α′ particles that form as a result of the low-temperature isothermal martensite in which the α′ particles are lath-shaped and significantly larger as shown in the optical micrograph in Fig. 4 of a sample cooled to –120°C and held for 10 hours. In these preliminary results, there is no evidence of either an amorphous phase, as suggested by Harbur [5], or the presence of a γ phase. We expected to observe an amorphous phase based on the similarity of this experiment to that of Harbur [5]. It is possible that the γ phase, as reported by Faure *et al.* [4], does form as an intermediate, but it is not retained to ambient pressure.

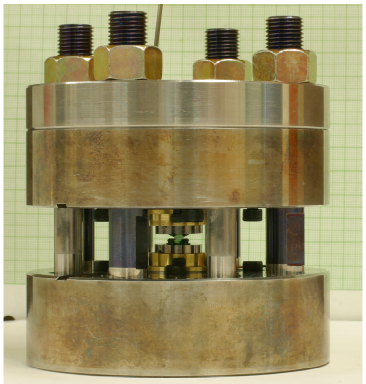

Fig. 1. Photograph of the large volume moissanite anvil cell.

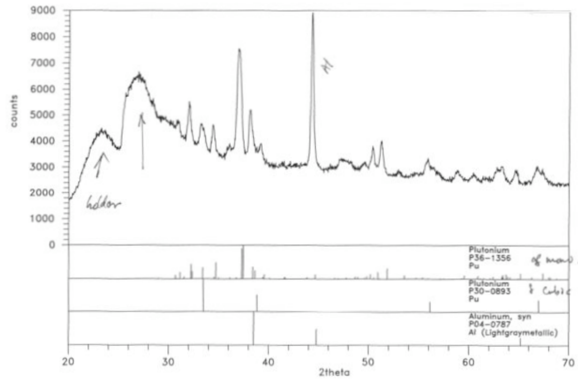

Fig. 2. X-ray diffraction scan of the recovered specimen.

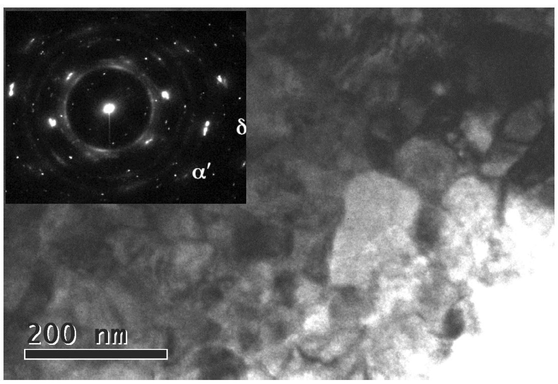

Fig. 3. Bright-field TEM micrograph and electron diffraction pattern of the compressed and recovered Pu–2 at.% Ga specimen. Very fine grains of the pressure-induced α′ phase and evidence of both the δ and α′ phases are observed.

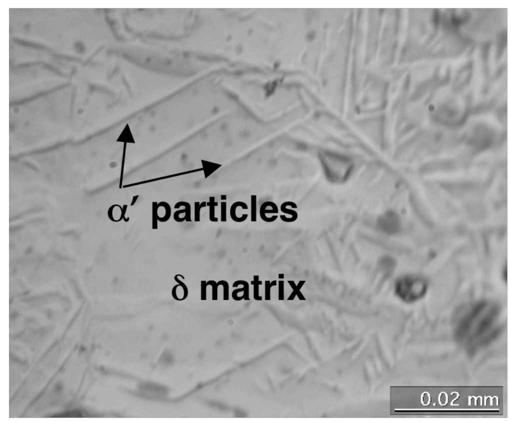

Fig. 4. Optical micrograph of the specimen that was cooled to –120°C and held for 10 hours. α′ particles are observed to be dispersed in the δ matrix.

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