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In- and ex-situ implantation of helium to characterise faults in titanium beryllide

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Background incl. aims

Transition metal beryllides, MBe_{12}, are an important material class in tokamak fusion reactors for use in tritium breeder modules as a neutron multiplier to maintain the refuelling cycle. During the neutron multiplying reaction chain, helium and tritium are also produced as well as knock-on radiation damage. Beryllium metal is a commonly used candidate neutron multiplier but develops large helium bubbles at breeder module operating temperatures which retain tritium on their inner surfaces (Zimber, et al., 2023), which complicates end-of-life recycling and would be a radiation and explosion hazard in an accident scenario. Titanium beryllide retains less tritium and is tougher (Kim, et al., 2021), so has been chosen as a candidate neutron breeder for EU-DEMO and JA-DEMO. There are still many material unknowns, however; what crystal defects develop, what do they do at high temperature, and how do helium and tritium/hydrogen behave in the microstructure?

Methods

In this study we used the University of Huddersfield's MIAMI-2 ion beam/TEM to implant 300kV He+ ions into 2µm thick sections ("bulk ex situ" sections for the purposes of containing the He peak) of TiBe12 at temperatures 387°C, 480°C, 600°C and 900°C to fluence of 10^{17} ions/cm². From this energy we expect a He peak approximately 900nm under the surface. We then re-sectioned the samples to produce cross sections parallel to the irradiation direction, with the He peak present as a horizontal band of helium approximately 900nm down the re-section. To begin investigating the effect of free surface proximity, a 100nm sample was also irradiated in-situ with 20keV He+ at 550°C. This produces a uniform low level of He (50x less than ex situ case) implantation, and a gently increasing comparable level of knock-on damage going down through the sample thickness.

Results

The thick "quasi ex situ" samples developed faults on {110} planes at temperatures up to 600°C; at 900°C the fault plane had changed to {111}. Displacement vectors were obtained using g.R analysis; some were found to be from the ½<110> or ½<011> families, some from neither previously observed displacement vector type (Banerjee, Jacobson, Zindell, & Mitchell, 1991). Bubbles of size 1-10nm were found at the He peak in samples irradiated at 480°C and 600°C. At 900°C, large bubbles (>200nm) evolved on grain boundaries and smaller (50-100nm) bubbles in grain interiors at the He peak (Sharp, et al., 2024). The {111} faults were preferentially associated with the grain interior bubbles, implying some kind of free surface or He sink effect at work.

The thin in situ sample contained some faults at room temperature before irradiation, which experienced very little change as the temperature was increased to 550°C. A high density of new faults began to appear almost as soon as He+ irradiation was started. Analysis of prior and irradiation induced faults in the in situ specimen is presented and compared with ex situ work to give an initial step to explaining the relationship between bubbles and faults.

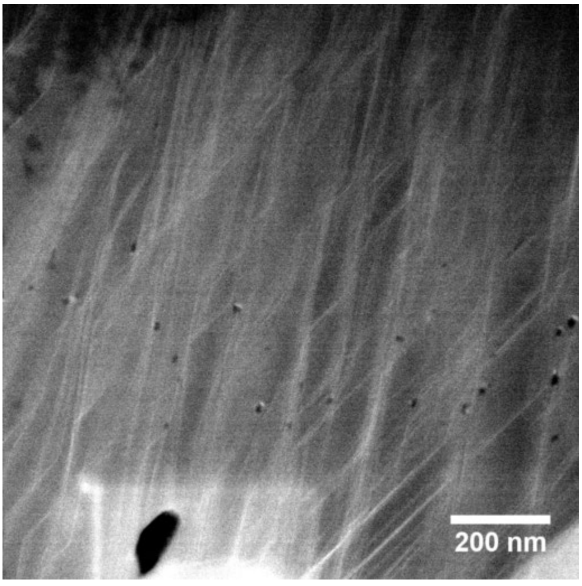
Conclusion

Planar crystal faults are present in unirradiated $TiBe_{12}$; new faults are created on irradiation with high energy (20-300keV) helium ions. At lower temperatures, faults lie on {110} planes, transitioning to {111} planes at some temperature between 600°C and 900°C in constrained material. In the same temperature interval, helium bubbles change from single nm size, to 50-100nm in grain interiors and larger on grain boundaries. Bubbles and {111} faults created at high temperature are preferentially associated.

Next steps in the subject will be to pinpoint the fault transition temperature, and to carry out and simulations and compare with experimental results to find the atomic configurations at {110} and {111} fault types, if there is a volume change between the two types that affects their relationship with bubbles, and whether/how they are sinks for interstitial helium atoms. The same process of understanding must then follow for hydrogen in the structure.

Image caption: ADF-STEM image from sample irradiated ex situ at 900C. White lines are {111} planar faults; small black objects are grain interior bubbles. At bottom is a larger bubble on a grain boundary. Image is part of a montage presented in Sharp et. al. 2024.

Graphic:



Keywords:

In-situ, irradiation, fusion, beryllide, defects

Reference:

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