Supplementary Material for:

Preparation of Binary and Ternary Laves and µ-Phases in the Ta-Fe(-Al) System for Property Analysis at the Microscale

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S1.1 Shaping the Samples

After synthesis with an arc melter, samples are obtained that have a relatively flat bottom and are otherwise convex. For 4 g sample weight, the dimensions are approximately 9-11 mm in diameter and 5-7 mm in height. For subsequent experiments involving correlative nanomechanical testing and electron microscopy, flat samples with a thickness of about 2 mm and parallel surfaces are needed. Several methods have been tested to obtain this sample geometry: Diamond wire cutting, electrical discharge machining (EDM), and grinding from the as-cast sample geometry.

During diamond wire cutting of the hard intermetallic phases, the amount of diamond wire used is very high due to the high hardness of the intermetallic phases which also results in long processing times of several hours to cut one sample. This process was therefore deemed unsuitable for the Ta-Fe(-Al) system.

During the non-mechanical EDM process, no direct deformation is introduced into the workpiece in contrast to other cutting tools. However, in case of brittle intermetallic phases, cracks and breakouts can increasingly occur due to the high internal stresses. Moreover, a recast layer forms on the sample surface that has to be ground off before further preparation. Due to the more prevalent cracking and breakout during EDM and the additional grinding step, this method was also considered inefficient and not well suited for the Ta-Fe(-Al) intermetallics.

To achieve the targeted sample geometry by grinding, the bottom side of the sample was ground down until it was completely flat. This is necessary to produce a flat sample with a parallel upper and lower surface. The abraded bottom side was then fixed to a

specimen holder with a movable center piece using crystal bond as temporary adhesive. Crystalbond 509 (Aremco) is suitable as a sample fixation because it can be easily applied and removed by heating and the applying temperature of approximately 150 °C for a few minutes has no influence on the high-melting intermetallic phases of the systems under consideration. The need to leave the prepared sample in acetone for several hours to remove other adhesives is thereby avoided. In addition, crystal bond offers a certain resistance to isopropanol, which was used for the metallographic preparation of the samples. The sample holder was used to facilitate handling of the comparatively small specimens during preparation, which will be discussed in more detail in section S1.2. To achieve the intended sample thickness of about 2 mm, the glued-on sample was machined with 500-grit SiC abrasive paper.

Due to the unavoidable mechanical processing of the sample surface, the two sample cutting methods presented first, diamond wire cutting and EDM, were classified as unsuitable for the TCP phases under consideration. Careful grinding down of the sample caused the least damage to the sample material in terms of the formation of cracks and breakouts.

S1.2 Metallographic Preparation

The entire metallographic preparation, i.e., grinding, polishing, and cleaning of the specimens, was carried out with technically pure isopropanol (99.9 %) as the basic lubricating, cooling, and cleaning medium. Cleaning in an ultrasonic bath was done after each step of the preparation to get rid of larger grinding or polishing particles from the previous preparation step. An overview of the individual preparation steps can be found in Figure S1. Starting with grinding the sample down to about 2 mm with 500-grit SiC abrasive paper, followed by grinding on progressively finer grit papers from 800-grit to 4000-grit. Each grinding step was done until no more scratches were visible from the previous coarser grit. The steps usually take between 5 and 10 min per grit, although experience shows that the time increases as the size of the paper becomes finer and more sheets of abrasive paper should be used due to wear.

After grinding and a thorough cleaning of the sample, polishing is required. Due to the long polishing times, this was done on a semi-automatic polishing machine. For this purpose, the sample was placed on the holder in the machine and loaded with a weight of approximately 1000 g. A solution of approximately 98 % isopropanol and 2 % polyethylene glycol 400 (PEG 400) served as lubricant. A perforated synthetic fiber cloth was used as the first polishing plate, as it has a high hardness and resistance to intermetallic phases. The first polishing step was done with a 3 µm grit (diamond particles) for about 5 to 6 h. After cleaning, the second polishing step was done on a silk cloth with up to 1 µm diamond particles for 6 to 8 h. The advantage of these parameters is the slow but steady specimen preparation, which allows existing scratches to be removed and breakouts to be reduced without further strong breakout of the brittle phases.

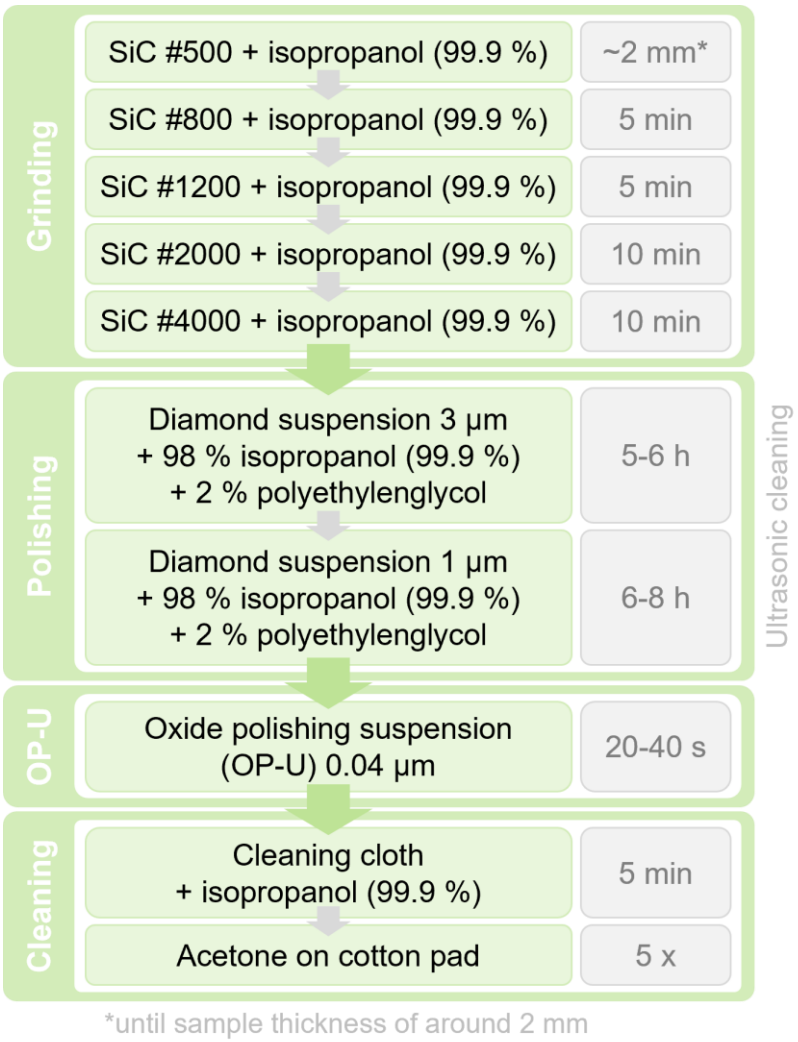

Figure S1: Metallographic preparation steps: Grinding, polishing, OP-U polishing, and cleaning. The processing tool, the medium used, and the duration are given for each step. Ultrasonic cleaning performed after each step is shown by the gray arrows.

The final polishing was carried out with an oxide polishing suspension (OP-U) with a particle size of 0.04 µm. Polishing was done for 20 to 40 s with the polishing suspension and water. The use of isopropanol was rather problematic here, as it jelled together with OP-U. After OP-U polishing, cleaning was performed on a separate disc with isopropanol. To determine whether the polishing time is sufficient, the specimen was viewed under an optical microscope with polarized light. If the preparation is sufficient, the microstructure should be slightly visible. Otherwise, another polishing with OP-U for another 20 to 40 s was done. For complete removal of the OP-U particles, a cotton pad is recommended, which is dripped with a little acetone and then carefully wiped over the sample. It is important not to use too much acetone, otherwise it will stain the surface.

After metallographic preparation, the sample must be thoroughly cleaned before it is mounted in a scanning electron microscope (SEM). The cleaning was done as before using isopropanol in an ultrasonic bath for several minutes. The sample can be detached by heating the crystal bond and then mounted on a SEM stub. For samples on which nanomechanical experiments are carried out, fixation on the SEM stub was again done with crystal bond, as this adhesive has a high stiffness and it must be

avoided that the adhesive material influences the test results. In addition, the sample is thereby fixed in such a way that even for longer measurements, such as EBSD maps, there is no risk of the sample slipping. However, since crystal bond is not conductive, silver glue was also used and the transition between the edge of the sample and the SEM stub was brushed with it.