1840 Microsc. Microanal. 28 (Suppl 1), 2022
doi:10.1017/S1431927622007243

Identifying Experimental Parameters for *In Situ* TEM Heating Experiments on Metastable Microstructures: Application to a Quasicrystal-Reinforced Al Alloy

B. Yavas[1*], M.X. Li[1], H.R. Leonard[1], S.P. Alpay[1, 2] and M. Aindow[1]

[1.] Department of Materials Science and Engineering, Institute of Materials Science, University of Connecticut, Storrs, CT, USA.
[2.] Department of Physics, University of Connecticut, Storrs, CT, USA.
* Corresponding author: baris.yavas@uconn.edu

Micro-electromechanical systems (MEMS)-based TEM specimen heating holders exhibit excellent thermal stability and minimal specimen drift, which allows thermally-activated processes to be studied dynamically at high spatial resolution. These advantages arise from the very small thermal masses of the samples used, and the samples can be FIB-cut specimens of specific phases or microstructural features. One of the main challenges in such studies is identifying appropriate experimental parameters. The choice of temperature for isothermal *in situ* heating experiments is critical if the key features of a thermally activated process are to be captured. At too low a temperature the process may not proceed to completion, whereas at too high a temperature the reaction or transformation may be so rapid that there is insufficient time resolution in the data captured, a least for conventional TEM experiments. When the samples are representative of bulk behavior, appropriate temperatures can be identified from ex-situ heating studies and/or non-isothermal calorimetry analysis. However, if the feature of interest is a local microstructural zone in a metastable state, those approaches are not feasible. Here we describe a survey "ramping" experiment which we have found to be particularly useful for identifying appropriate temperatures for isothermal *in situ* heating experiments. The temperature profile for a typical experiment of this type is shown in Figure 1. This particular experiment was designed for use on metastable microstructures in aluminum alloys where the transformations of interest occur in the solid state. The ramp involves holds at 100 ˚C for 3 min to drive off any adsorbed moisture, and at 350 – 500 ˚C in 25 ˚C increments for 10 min to observe the processes occurring at each temperature.

To illustrate this we present data from laser surface treated samples of a Al-Cu-Fe-Cr alloy, which contains a dispersion of icosahedral quasicrystals; these hard dispersoids serve as reinforcements in the FCC Al matrix giving a composite strengthening. The laser studies were performed to evaluate the potential for using this alloy in additive manufacturing. Since the quasicrystal reinforcement phase is inherently metastable [1], it is important to understand the thermal decomposition (crystallization) of this phase, and *in situ* heating studies can provide unique insights. Specimens for these TEM experiments were prepared from laser tracks on the alloy surface using the FIB-based technique developed by Vijayan *et* al. [2]. All thinning steps were performed using standard FIB lift-out techniques before transferring the specimen to the MEMS-based heating holder chip while minimizing the ion beam damage and Ga contamination. The heating experiments were performed on an FEI Talos F200X STEM using an FEI Nano-Ex/iV heating stage with an external Keithley temperature controller [3]. The progress of the thermally activated transformations was recorded by capturing bright field (BF) STEM images at 1s intervals throughout the experiment. A selection of these images is shown in Figure 2. To reveal the chemical redistribution associated with the microstructural changes, high-angle annular dark field (HAADF) STEM images and EDXS spectrum images (Figure 3) were acquired at room temperature before and after the survey experiment under the same conditions. Based upon these data, two main processes were identified: a redistribution of the Cu segregated around the quasicrystals into discrete intermetallic particles starting at around 350 ˚C, and a crystallization of the quasicrystals themselves at 450-475 ˚C. While these transformation temperatures will clearly be affected by the prior holds at lower temperatures in the ramp, such observations do provide a useful guide to the selection of temperatures for isothermal observations of transformation kinetics [4].

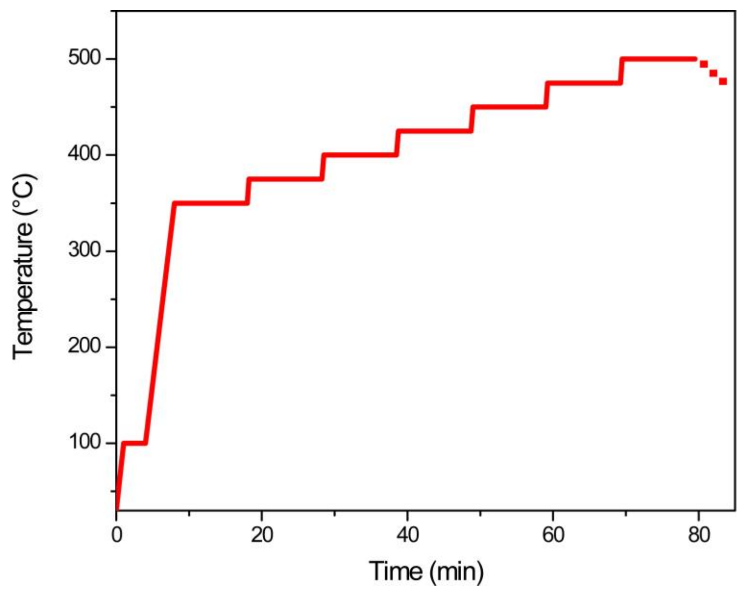

Figure 1. Temperature profile for the survey ramping experiment

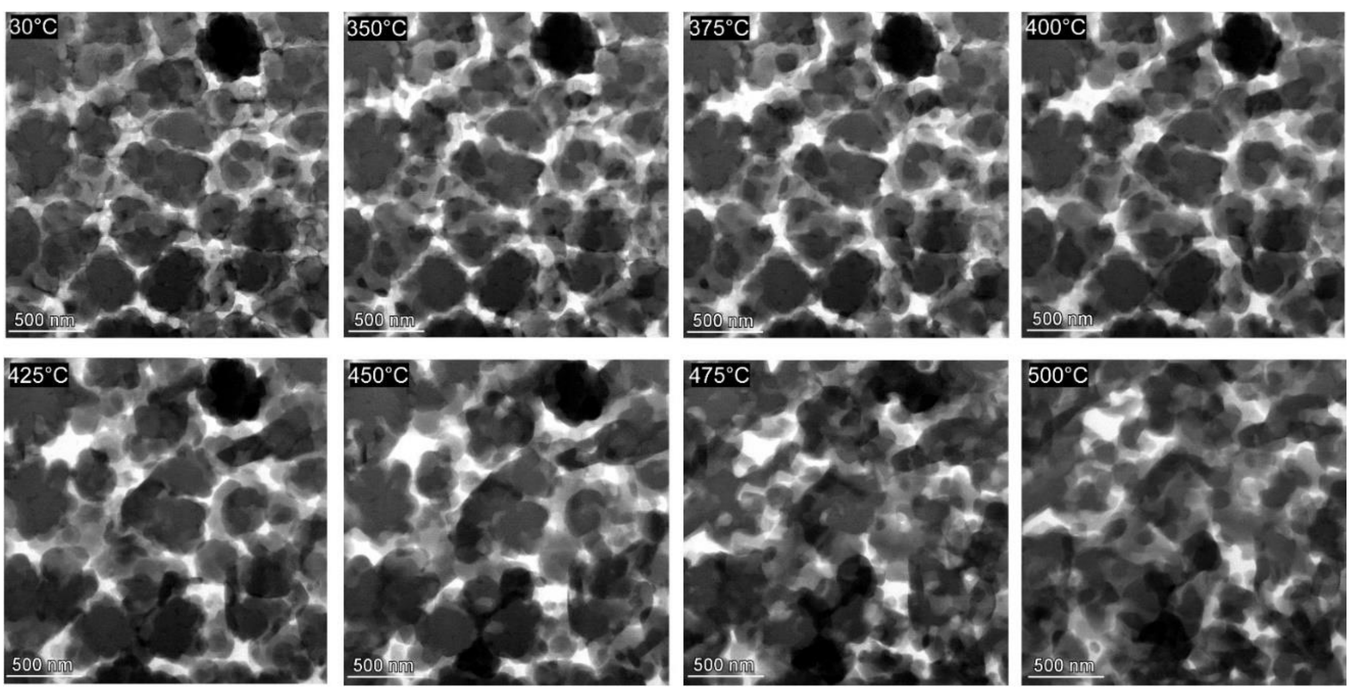

Figure 2. A series of BF STEM images from the ramping *in situ* heating experiment from 30°C to 500°C

https://doi.org/10.1017/S1431927622007243 Published online by Cambridge University Press

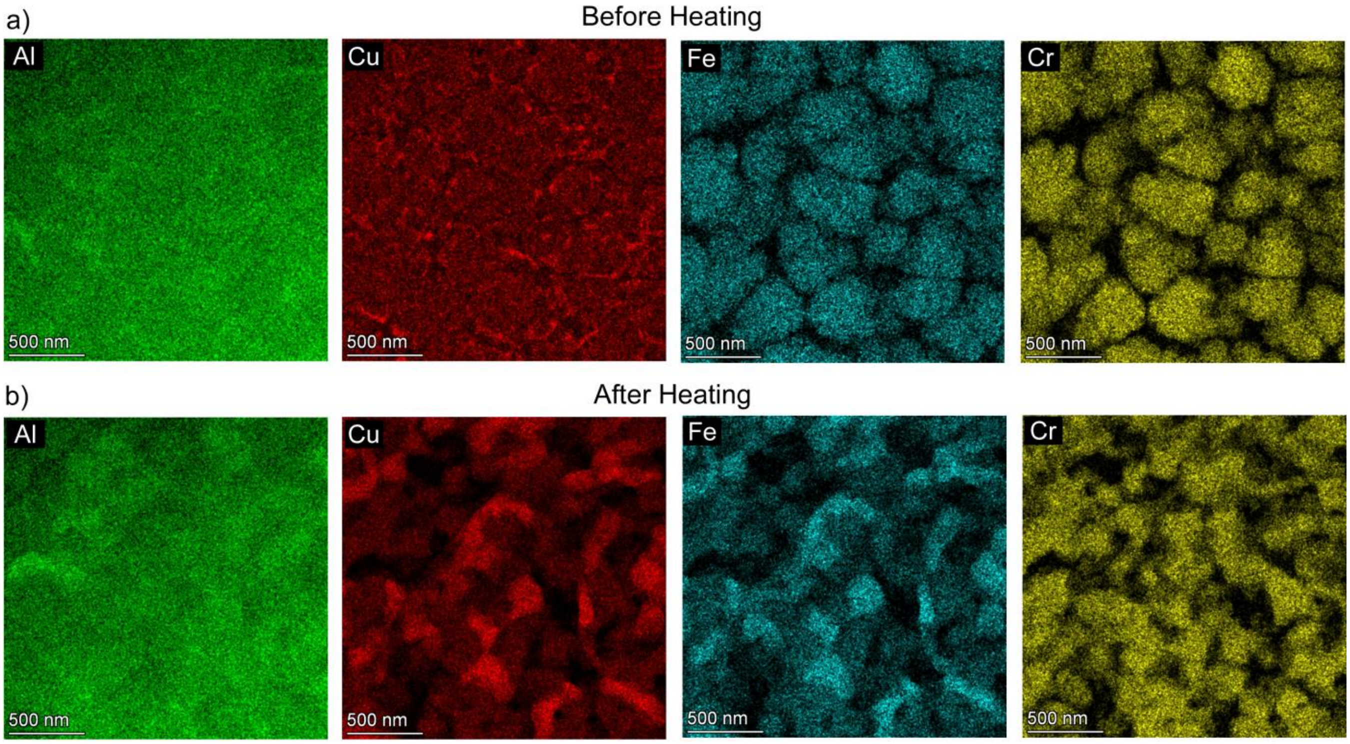

Figure 3. STEM EDXS intensity maps from the region before (a) and after (b) *in situ* heating

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[4] These studies were performed in the UConn/ Thermo Fisher Scientific Center for Advanced Microscopy and Materials Analysis (CAMMA).