Multiscale structure measurement for Al-Li alloy

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1. Introduction

The iMATERIA (BL20) is equipped with small angle and backward detectors suitable for the multiscale structure measurements. Last year, we reported that a simultaneous measurement system of both small angle scattering, and wide-angle diffraction with high resolution could be constructed by using our originally developed neutron high-absorption glasses as an optical slit for the neutron beam. This system is capable of continuously measuring 10 samples by using a sample exchange device. The measurement temperature is, however, limited to room temperature.

The purpose of this study is to extend the measurable temperatures beyond room temperature. A new electric furnace with low heat capacity and another optical slit from neutron-absorbing glass were prepared. The furnace is capable of rapid heating and cooling, which is suitable for measuring structural changes in a short period of time. Multiscale microstructural changes in Al-Li alloy over time were investigated to verify the usefulness of the new measurement system.

2. Experiment

The furnace was designed to be as compact as possible, and a sheathed heater was used to reduce the heat capacity. The heater temperature is controlled by a PID system, with a maximum temperature of 350°C. The sample temperature is monitored by the lattice constant of the sample or the aluminum cover sheet for the powder sample obtained from diffraction data. Photo. 1 shows the arrangement of the glass slit and the electric furnace. The neutron beam enters from the left side. The beam size is 12 mm. Backscattered diffraction from the furnace is effectively suppressed by the glass slits.

The Al-Li alloy was solution-treated at 560°C for 1 hour, then quenched in ice water. The sample size is 20mm in diameter and 2mm thick. The sample was measured at room temperature for 1 hour, then the temperature was increased to 150°C for 12 hours to measure the change in structure over time.

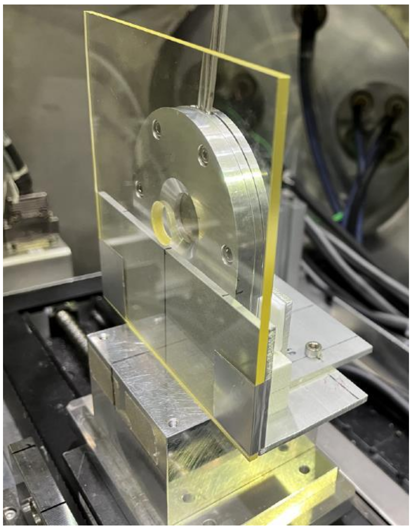

Photo. 1 Arrangement of glass slit and electric furnace.

3. Results

Small angle scattering, and diffraction profiles of Al-Li alloy are shown in Fig. 1. The small angle scattering profiles at room temperature show the presence and distribution of nanoscale precipitates. The diffraction profiles indicate that the precipitate is Al_3Li. The growth of nanoscale Al_3Li due to aging at 150°C was clearly detected in both small angle scattering, and diffraction profiles. The measured data were accumulated hourly. As seen in Fig. 1, significant microstructural changes are observed immediately after heating. Therefore, the collected data were divided into every 5 minutes. Figure 2 shows the small angle scattering profiles for the initial 2 hours of aging. The iMATERIA measurements confirmed that quantitative small angle scattering data were available within 5minutes. The small angle scattering profiles at room temperature and at 150°C for the first 5 minutes are identical. The time variation of the sample temperature was examined from the lattice constant. The diffraction data were divided every minute. The results are shown in Fig. 3. Open red circles in the figure correspond to the 5-minute data. The sample temperature reached 150°C at 10 minutes after heating. On the other hand, the average temperature during the first 5 minutes was around 90°C, suggesting that aging was not effective during the first 5 minutes.

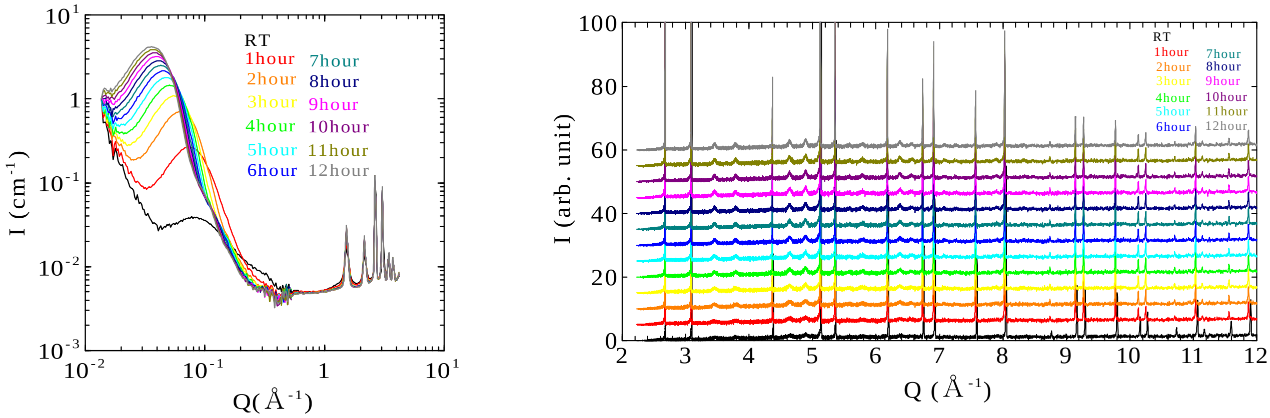

Fig. 1 Time dependence of simultaneous small angle scattering, and diffraction profiles of Al-Li alloy.

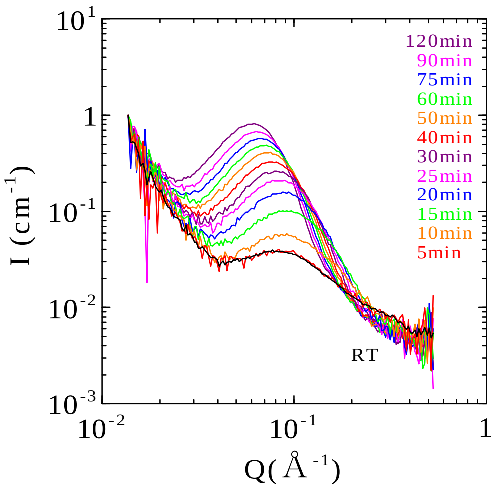

Fig. 2 Small angle scattering profiles in smaller time division.

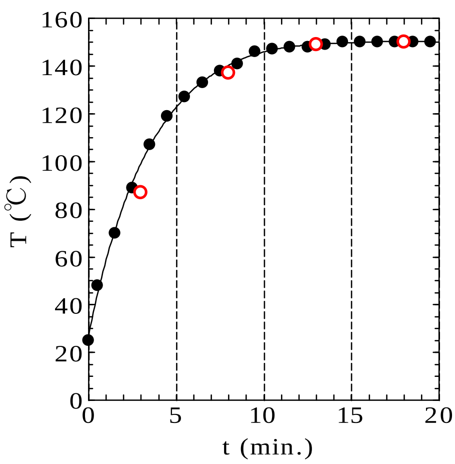

Fig. 3 Temperature variation of the sample as a function of heating time.

4. Conclusion

The originally developed electric furnace can handle relatively rapid structural changes, because it reaches the set temperature in about 10 minutes. Simultaneous small angle scattering, and wide-angle diffraction measurements were validated for time-dependent multiscale structure using this furnace, and multiscale structure measurements of Al-Li alloy revealed the origin of precipitates and their growth at 150°C.