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Development of in-situ DSC-SWAXS system for metallic alloys - a preliminary test

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Introduction

Diffusional phase transformation process in multi-component alloy systems is one of the major research subjects in the development and control of microstructures in metallic or oxide materials. In order to examine microstructural stability of glass materials, understanding crystallization process of the glassy materials and supercooled liquids is quite important. However, the stability of these glasses or liquids depend on the thermal hysteresis or environments such as trace impurity of oxygen etc. Therefore, we need an in-situ and simultaneous measurements of thermal properties and structural properties in order to obtain reliable relationship between relaxation / glass transition/ crystallization or precipitation processes. For lower temperatures with moderate sensitivity, commercial DSC/SAXS cell is readily available. However, these products are not suitable for the phase transition of metallic glass where the change in heat capacity is rather small and the temperature should be higher. Therefore, we have attempted to develop an in-situ DSC/SWAXS system applicable to experiments on phase transformation processes in metallic materials.

Experimental

The in-situ DSC/SWAXS apparatus developed in the present proposal has several points that are different from ordinary commercial DSC-SAXS cells. One is that the DSC cell part is a modified version of commercial high-sensitivity DSC, originally designed for inorganic materials up to about 800 K with enough sensitivity to evaluate glass transition temperature of bulk metallic glasses. To maintain sensitivity and high-temperature use, the DSC cell was designed to be used in the vertical alignment. Therefore, it requires that the X ray should be introduced from bottom window, goes through the thermopile units and DSC pan, then the direct /scattered and diffracted beams go out of the upper window. Two one-dimensional PSPCs are to be used in the present system, one for SAXS and the other for WAXS. The DSC unit is leak-tight and can be evacuated during the scan. At the beam-line 15A, the incident beam was diffracted upward with a Ge 111 flat single crystal wafer. Diffraction condition of Ge 333 was used to orient the beam upwards. Since the wavelength of the incident X-ray is 0.15 nm at BL15A, the diffracted beam, along with the offset angle given by a front mirror, is ideally vertical in the present condition.

Figure1 gives a photograph of the DSC/SWAXS system used in the present test. The stage was installed close to the focal point of the incident beam to produce a small diffracted beam.

Present test revealed that some improvement on the DSC cell structure was necessary for quick setup of the apparatus in this crowded beamline. For this purpose, a new configuration of thermocouples with larger angle of acceptance in DSC cell is now under fabrication, and planned to be used for the in-situ experiment in the second term of 2006.

References

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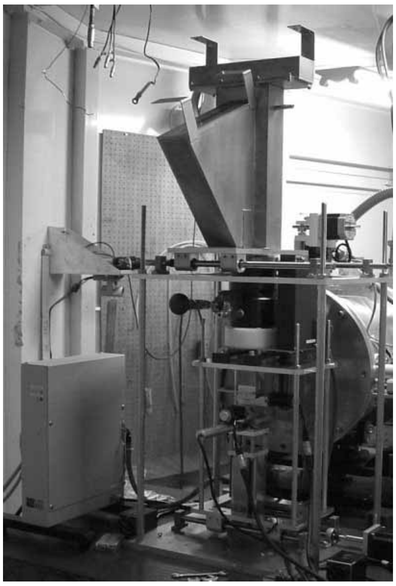

Fig.1 Photograph of the in-situ DSC-SWAXS apparatus installed at the back end of BL-15A. The incident beam is diffracted vertically upwards, goes through the DSC cell and then enter the SWAXS chamber.