

Compact High-Temperature Shear-Cell Furnace for In-Situ Diffusion Measurements

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Abstract. A compact-sized shear cell for *in-situ* interdiffusion measurements in liquid alloys at high-temperature is presented. It is designed for being used with X-ray radiography in microgravity environment and also in ground-based experiments. Within outer dimensions of 40x40x20 mm, the shear cell contains six samples in long capillaries which remain spatially separated during melting, homogenisation and optional μg -injection. The diffusion is then initiated by shearing both material samples together. Operating temperature is as high as 1500 °C. Stable temperature distribution is found and no sample leakage occurs. The thin layout of the shear cell results in a good X-ray contrast supported by making use of graphite heating foils. First results of ground experiments are presented.

1. Introduction

The measurement of diffusion coefficients in liquid alloys faces a number of experimental challenges of which flow effects during melting of the diffusion couple, buoyancy-driven convection while diffusion annealing, and the formation of shrinkage holes as well as microstructure formation during solidification are the most prominent [1, 2]. For a few sample systems these effects have small influence on the results in standard long capillary experiments, particularly if carried out in μg . An already successfully applied platform for such experiments in μg is the ATLAS-M [3] experiment on the sounding rocket MAPHEUS [4]. For other sample systems the measurement may be noticeably disturbed by the mentioned effects. The challenges to reduce the influence of these can be met by combining a shear-cell technique with X-ray radiography [5, 6] under μg -conditions.

X-ray radiography (XRR) allows time-resolved *in-situ* experiment which are unaffected by microstructure formation and shrinkage holes. It requires though sufficient X-ray contrast in the sample system and a flat and thin furnace design consisting of only weakly X-ray absorbing materials. Within a shear cell the two parts of a diffusion couple are melted separately and homogenized before the diffusion measurement is started by shearing both parts together. Therefore flow effects and segregation of higher melting point phases during heating do not affect the measurement [7]. Furthermore, the diffusion time is well known. To maximize the number of diffusion pairs which can be processed during a flight of the sounding rocket, neighbouring diffusion pairs share one sample material. This allows to investigate a maximum of six diffusion couples simultaneously. Since buoyancy-driven convection poses problems in ground based experiments, the μg -environment on the sounding rocket MAPHEUS is used for

the measurement of reference values. MAPHEUS offers a μg -time of up to 4 minutes and can therefore be used for fast diffusing samples such as Ge-based or Al-based alloys. For the operation on the sounding rocket, the shear cell employs a compact, light weighted and sufficiently rugged design.

2. Shear-Cell Furnace Characteristics

The cuboid shaped shear cells' overall dimension count 40x40x20 mm. The crucible is entirely made out of graphite. Its high thermal conductivity is a key for a constant temperature regime over the samples. Further graphite is fairly inert with a large number of sample systems at moderate temperatures. For other more reactive systems graphite is a good choice for rapid diffusion experiments enabled by XRR when the chemical reaction between graphite and melt is slow. The chosen sample size of thin rods with 1.5 mm in diameter and 15 mm in length corresponds to previous diffusion experiments that have been performed in classic long capillary experiments. As a result, six different diffusion couples can be measured at the same time and temperature, restricted by the condition that a diffusion couple contains one sample from each of its neighbours. This graphite crucible is a set of two components. The upper part

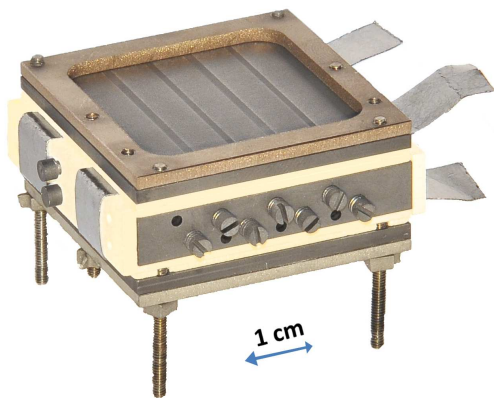


Figure 1. Photography of the assembled shear cell

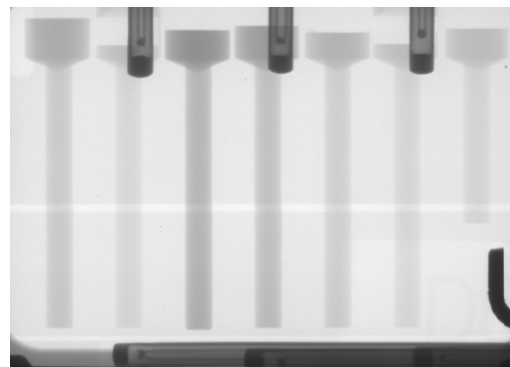


Figure 2. X-ray image showing configuration of the shear cell with liquid $Al_{1-x}Ni_x$ ($x=0..12.5$ at%) homogenized shortly before shear.

contains half of each capillary as well as the reservoir to compensate volume changes during melting and tolerances in the manufacturing process. The reservoir is sealed by a graphite piston which in turn is driven by some graphite felt compressed with graphite screws. The lower part contains the matching other half of each capillary in the form of a blind hole. Both elements put together deliver seven independent containers, each sealed and with congruent cross section at the separation plane. There is a lateral distance of 4 mm between neighbouring capillaries. The lower part is meant to perform the shear movement and therefore runs in a U-shaped rail preventing any lateral movement. From beneath it is supported by another set of graphite felt to pressurize the separation plane within the divided crucible. By this, leakage is inhibited and at the same time shear movement is allowed. The temperature is measured with three pairs of thermocouples (TCs) being positioned on the left, in the centre and on the right of the shear cell as shown in figure 2. Sheathed TCs of diameter 1.5 mm are used. Each two of them are arranged in a straight line between two samples on upper and lower end to confirm constant temperature. For low temperature operation ≤ 1100 °C Type-K TCs are currently in use whereas for temperatures up to 1500 °C Type-S TCs will be used. To enable compensation of any arising gradients in temperature along the diffusion couples, the shear cell is equipped with

two independently controlled heating zones, one covering the top and the other one the bottom part. The material chosen for the heaters is pure expanded graphite foil of 0.15 mm thickness and has the ability to cope with high rates of heat flux. Further, it shows negligible X-ray absorption which is beneficial for the *in-situ* XRR experiments. Each heater provides 750 W of heating power. To electrically insulate the heater from the crucible the heaters are placed between thin layers of alpha boron nitride. The heater covers both sides of the crucible and passes over at the short end. Oppositely the heaters are linked a to power junction by niobium connectors. The shear cell is sandwiched between additional layers of bulk graphite and frames of niobium connected among each other by six 2 mm-rods. By that this shear cell gets rugged and prepared for sounding rocket environment. It is held in place by four of the niobium rods acting as its legs, reducing the local heat discharge to a minimum amount. The entire setup is placed within a thermal insulation of niobium-reflector and aluminiumoxide-fibres in an aluminium-box, each fitted with windows for the X-ray beam. The experiment takes place in high vacuum. The shear movement is regulated by an external servo drive controlled by a microcontroller-based electronics unit. The movements are transmitted by a wire of molybdenum to the crucibles shifting lower part.

3. Experimental Results

Several test experiments have been carried out where the overall functionality of the shear cell has been demonstrated. The maximum set temperature in first tests has been 1100 °C requiring a heating power of about 240 W to hold this temperature. At a setpoint of 900 °C a spatial temperature gradient of approximately 1 K in samples is observed. The heating rate is about 2 K/s at 900 °C, when the heating power is limited to 375 W. First experiments on aluminium-alloys proved *in-situ* X-ray functionality as well. Figure 3 shows a grey value profile of $\text{Al}_{95}\text{Ni}_5$ vs. $\text{Al}_{85}\text{Ni}_{15}$ obtained by using the presented shear-cell setup. The difference in Ni-concentration of 10 at% is resolved with about 500 units in grey value. By this method profiles for diffusion couples with only 2,5 at% differences in Ni-concentration can be analysed. To gain diffusion profiles from these data, the measured grey values at each position have to be normalized [6]. Then diffusion profiles following the expected error function as a solution of Fick's law of diffusion are obtained. For the measurements a microfocus X-ray source with 100 kV and up to 10 W

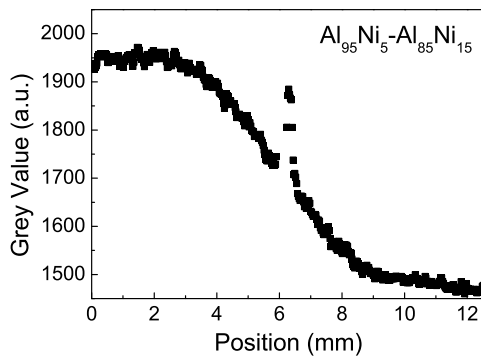


Figure 3. Gray value profile of the diffusion couple $\text{Al}_{95}\text{Ni}_5$ vs. $\text{Al}_{85}\text{Ni}_{15}$ as obtained using the presented shear-cell setup without normalization. The peak around 6 mm results from the necessary slit between the U-shaped rail and the upper part of the crucible as can be seen in fig. 2.

in combination with a scintillation detector was used. The detector frame rate was set to 1 Hz, which gives good contrast-properties. To reduce artefacts caused by X-ray scattering, the beam was collimated by an aperture with a diameter of 0.6 mm in front of the X-ray source. Sample-source-detector distances were set such that a total resolution of 29 μm corresponding to sample plane was achieved. Further more extensive tests of the shear movement were carried out. First the cell was tested and calibrated in cold state. In addition it also proved its functionality in a

900 °C Al-Ni experiment. However, the reliability in the precision of the shear movement which is important to obtain correct results [8] turned out to require further development. Incidents of leakage of the sample-material at the shear front did not occur. The whole setup has been already successfully tested for flight readiness in a shaker test performed at the ZARM-Centre in Bremen, Germany.

4. Conclusion

A novel shear cell for the use with X-ray radiography on a sounding rocket has been designed, successfully tested, and space qualified. It allows the simultaneous measurement of six diffusion couples at the same temperature. The shear-cell furnace offers an excellent X-ray contrast. Pressure on reservoirs is adjusted such that neither free sample surfaces nor sample leakages occur. The shear cell is designed for temperatures of up to 1500 °C which will enable to measure higher melting point alloys as Si-rich Si-Ge and Ni-rich Al-Ni in future.

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