A Shear cell technique with thin samples for ground based liquid diffusion measurements

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Abstract

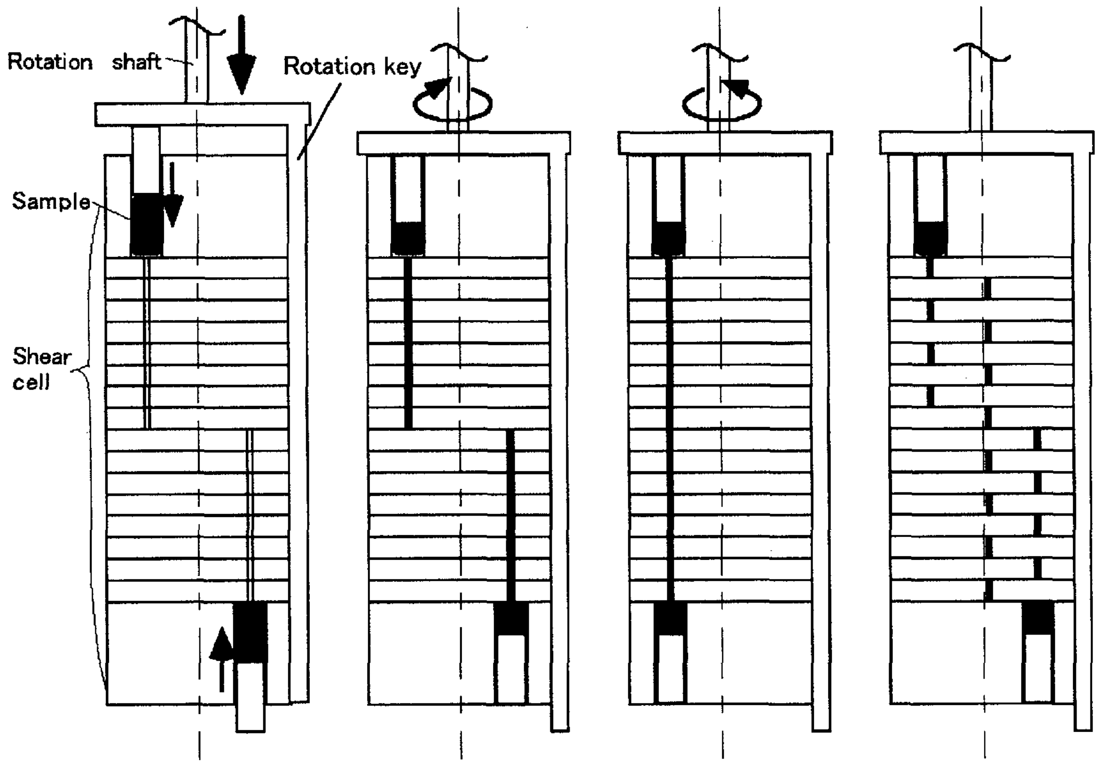
It is hard to measure liquid diffusion coefficients on the ground because of the buoyancy convection. One way to suppress the convection is to reduce the sample radius. We developed a shear cell with a casting system for thin samples and carried out a diffusion experiment by the shear cell technique. Samples were cast into 0.5 mm diameter capillaries successfully. The samples were successfully divided completely at the upper side, but the samples at the lower side were not divided because the rotation key bent at high temperature. It was found that the rotation key should be a rod with larger strength, and that the shear cell experiments with 0.5 mm diameter samples can be carried out.

Introduction

The diffusion coefficients of molten metals and semiconductors are important properties both for industries and for liquid theories. But it is hard to measure the liquid diffusion coefficients on the ground because the buoyancy convection is hardly avoided. One way to suppress the convection is reducing the sample radius; the flow in liquid is suppressed by the crucible wall. Diffusion experiments using 1.5 mm diameter samples were carried out on the ground. It was found that the diffusion coefficients obtained from the experiments seemed to be larger than the estimated value and that the sample diameter should be less than 1 mm. However, it is hard to make and treat samples with a diameter less than 1 mm because the thin samples can be easily broken. We developed a shear cell in which liquid samples can be cast into 0.5 mm diameter cylinder followed by a shear cell diffusion experiment. A diffusion experiment using the shear cell technique was carried out to confirm its performance.

Development of the shear cell

Figure 1 shows the schematic drawing of the shear cell with casting system for thin samples. The sample materials are put in pouring basins at the upper and the lower side of the shear cell. After the sample materials are melted, they are cast into the capillaries by pushing the rotation shaft. When the temperature is stabilized at the diffusion temperature, the separated diffusion couple is joined by rotating the rotation key combined with the rotation shaft, and the experiment starts. The sample is divided into some pieces at the end of the experiment and these small pieces are solidified.



(a) Samples are put in pouring basins at room temperature

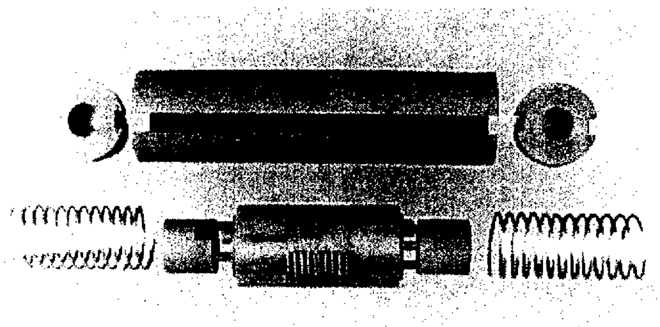
(b) Samples are cast into 0.5 mm diameter capillary over the melting temperature

(c) Samples are joined at the diffusion temperature. A diffusion experiment starts.

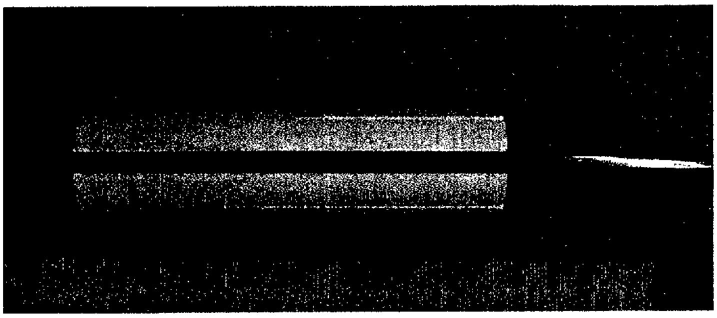
(d) Samples are divided into small pieces at the end of the diffusion experiment.

Figure 1 Schematic drawing of the shear cell with casting system

Photograph 1 shows the shear cell, which consists of 16 disks of 1.5 mm thickness. It was made of graphite, which has excellent mechanical and lubricant properties even at high temperature. Three samples can be set in the shear cell and three experiments can be performed at once. Photograph 2 shows the shear cell set in the outer tube and the rotation shaft made of tantalum, which has the high melting temperature. The rotation key combined with the rotation shaft, which rotates the shear cell, is also made of tantalum.



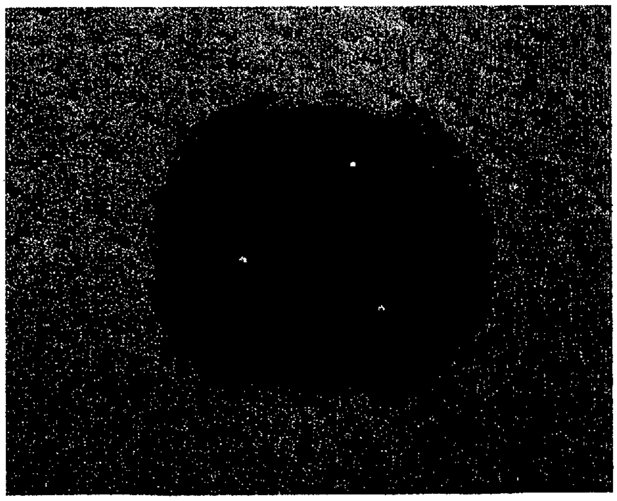
Photograph 1 Shear cell with casting system made of graphite



Photograph 2 Shear cell setting (rotation shaft attatched to shear cell)

Shear cell experiment

A diffusion experiment of Ag and $Ag_{0.95}Au_{0.05}$ was carried out with the present shear cell. Figure 2 shows the shear cell cartridge using a Large Isothermal Furnace in NASDA. The diffusion temperature was 1373 K and the diffusion period was 6 minutes. From the observations after the experiment, it was confirmed that the samples were cast into the 0.5 mm diameter capillaries successfully. The samples at the upper side were divided into small pieces as shown in Photograph 3, but the samples at the lower side were not divided completely because the rotation key bent at high temperature. It was



Photograph 3 Disk of shear cell after the experiment (silver color circles are divided samples of 0.5 mm diameter)

considered that the torque became larger than an estimated value because of the weight of the rotation shaft. And also, the rotation key outside of the shear cell was exposed at high temperature, at which the strength of tantalum was smaller than estimated. It was found that the rotation key should increase its strength, and then the diffusion experiments with 0.5 mm diameter samples could be performed successfully.

Conclusion

The shear cell with casting system for thin sample was designed and tested. The samples were cast into 0.5 mm diameter capillary in the shear cell successfully. A part of the samples couldn't be divided at the end of the experiment due to the bend of the rotation key at the high temperature. It is conclueded that the design of the rotation key should be changed.

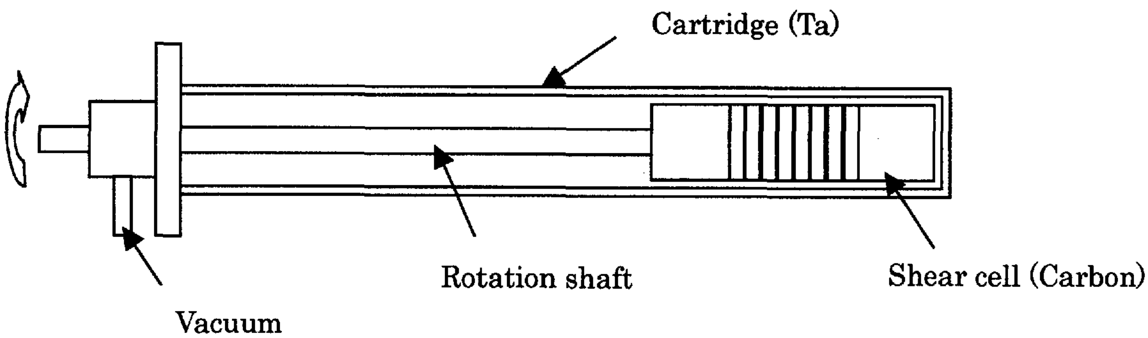


Figure 2 Shear cell cartridge using a Large Isothermal Furnace