X-ray diffraction measurement at 0.20K

S.Naher [a,1], H.Suzuki [a], M.Mizuno [a], Y.Xue [a] and H.Fujishita [a]

[a] *Department of Physics, Kanazawa University, Kakuma-machi, Kanazawa, 920-1191, Japan*

Abstract

We have developed an x-ray diffraction measurement system for powder samples below 1K. We use a dilution refrigerator (D.R) of Oxford Instr. Kelvinox VT, which was modified for the x-ray measurement. The x-ray beam was reduced approximately 1/12, after passing through the windows of the dilution refrigerator. The windows are consist of 4 walls of Be 2mm thick, 2 Al film 10μm thick and 2 aluminized mylar walls. The lowest temperature of the x-ray measurement was about 0.20K. We have studied the temperature gradient between the specimen and the thermometer (RuO_2) which was attached to the mixing chamber. The results of our measurement suggest that there is no temperature difference between the RuO_2 on the mixing chamber of D.R. and the specimen down to 0.5K. Below this temperature the gradient was observed to some extent.

Key words: X-ray Diffraction; Dilution Refrigerator

1. Introduction

Many interesting materials show phase transition at low temperature below 1K. To investigate these phase transitions and the ground states of these materials requires measurement of the physical properties below 1K using a ^{3}He-^{4}He dilution refrigerator. The crystal structure may be the most essential information to understand the new phase below the transition temperature. Low temperature x-ray diffraction measurement, even in the temperature range of 1.5K, however, is done very seldom. Here we show an x-ray diffraction measurement system for powder sample below 1K [1]. The system is composed of the ordinary x-ray appratus and the ^{3}He-^{4}He dilution refrigerator (D.R).

2. Experimental Results and Discussion

The dilution refrigerator was the Oxford Instr. Kelvinox VT, which was modified for our x-ray measurement system and mounted on a two-circle gonimometer, provided by Rigaku Co. The cooling power of the dilution refrigerator is 25μW at 100mK and the base temperature is 30mK. The x-ray diffractometer was a Rigaku RINT2000. The condition for the production of the x-ray beam was 45kV and 300mA with rotating Cu target. The x-ray beam, which passed through 4 walls of Be and 4 aluminized mylar walls as shown in Fig. 1, was reduced to approximately 1/100. With the x-ray beam produced under the condition of 45kV, 300mA, the lowest temperature of the dilutions refrigerator was about 0.18K. The heat input due to the x-ray beam for our cryostat was estimated as follows: x-ray beams, 45kV-300mA, 30kV-200mA and 32kV-150mA the corresponding heat input was 84μW, 34μW and 19μW, respectively. The powder samples were mounted on a gold plated sample holder made of oxygen free high conductivity Cu, and the groove size was about 20×12×2mm. The picture of the sample holder with carbon resistance is shown in fig. 2. The

[1] E-mail: shumsun@nihonkai.kanazawa-u.ac.jp, Present address: Department of Physics, Kanazawa University, Kakuma-machi, Kanazawa, 920-1191, Japan

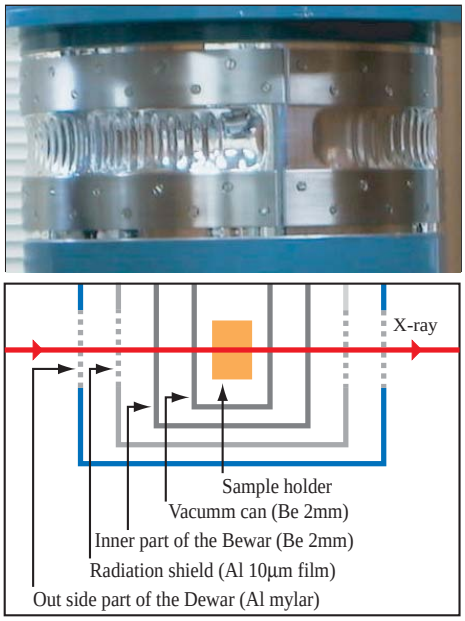

Fig. 1. Part of the dilution refrigerator for passing x-ray. The window of the radiation shield was replaced by 10μm Al film.

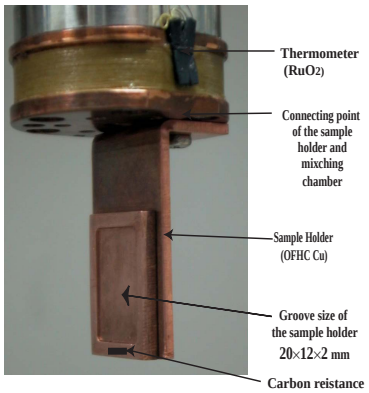

Fig. 2. Picture of the sample holder with carbon resistance.

sample holder is connected with the mixing chamber of the dilution refrigerator by screw. For the improvement of the thermal conduction between the sample holder and the mixing chamber we used the apeazon N grease between the mixing chamber and the sample holder. A RuO_2 thermometer was attached to the mixing chamber. We measured the temperature of the specimen by using this thermometer. We studied the temperature difference between the specimen and the thermometer by measuring the Jahn-teller distortion of $TmVO_4$ [1]. The result suggests that we are able to measure the x-ray diffraction below 1K properly.

After this we have improved our measurement system, by replacing the aluminized mylar film radiation shield with 10μm Al film, which was placed between the Outer Vacuum Can (OVC) and the nitrogen bath. Under this condition the x-ray beam was reduced approximately 1/12, after passing through the windows of the dilution refrigerator. The part of the dilution refrigerator for passing x-ray is shown in fig. 1. With the x-ray beam produced under the condition of 45kV, 300mA, the lowest temperature of the dilutions refrigerator was about 0.20K. We worried about the temperature difference between the specimen and the mixing chamber.

We investigated the temperature difference measured by using a carbon resistance thermometer of 100Ω. This resistance was attached to lower part of the sample holder by the GE varnish. To prevent the ohmic self heating we used a small current of ∼μA. The carbon thermometer was calibrated against a calibrator RuO_2 thermometer on the mixing chamber without the x-ray beam. Then we measured the temperature change of the sample holder with the x-ray beam on and off at the temperatures between 0.2K and 1K.

The results show that there is no significant temperature difference between the mixing chamber and the thermometer on the sample holder down to about 0.5K. Below 0.5K the deviation of the temperature was observed to some extent.

Then in our x-ray measurement below 1K, we measure the temperature of the sample by using a carbon thermometry on the sample holder.

References

[1] H.Suzuki, S.Naher, T.Shimoguchi, M.Mizuno, A.Ryu and H. Fujishita, J. Low Temp. Phys. 128, 1/2 July (2002) to be published.