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2007 J. Phys.: Condens. Matter 19 425203

(http://iopscience.iop.org/0953-8984/19/42/425203)

IOP PUBLISHING JOURNAL OF PHYSICS: CONDENSED MATTER

J. Phys.: Condens. Matter **19** (2007) 425203 (5pp) doi:10.1088/0953-8984/19/42/425203

The effect of pressure on the nuclear quadrupole resonance and ruby fluorescence in a NiCrAl pressure cell

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Received 3 August 2007
Published 18 September 2007
Online at stacks.iop.org/JPhysCM/19/425203

Abstract

A pressure of 4.0 GPa was achieved using a piston–cylinder-type NiCrAl pressure cell which possesses a large sample space of 4.4 mm diameter × 15 mm length at ambient pressure. As monitors of the pressure, the ^{63}Cu nuclear quadrupole resonance (NQR) of Cu_2O and ruby fluorescence were studied at the same time. The relation between the NQR frequency and the R1 line of the ruby fluorescence was investigated at pressures below 3.2 GPa.

1. Introduction

The role of pressure has become increasingly important in strongly correlated electron systems, because a number of 3d transition metal oxides, heavy fermion systems, and organic conducting systems exhibit a variety of phases including a superconducting (SC) phase upon applying pressure. In the case of pressure-induced superconductivity, a spin- or charge-ordering phase often appears as a neighboring phase on the lower pressure side; a spin density wave (SDW) and an antiferromagnetic ordering (AFO) appear in the low pressure region for organic conducting systems and Ce compounds, respectively. The 3d transition metal oxides, a spin-ladder cuprate $Sr_{14-x}Ca_xCu_{24}O_{41}$ [1, 2], and the β-bronzes AV_6O_{15} (A = Li, Na, and Ag) [3, 4] also exhibit pressure-induced superconductivity. In the spin-ladder cuprate, the appearance of a charge density wave (CDW) was suggested from dielectric constant and resistivity measurements for the Ca-poor region of $x \leqslant 10$. Superconductivity was observed in the Ca-rich region of $x \geqslant 10$ on applying pressure of over 3 GPa [5]. In the β-bronzes, pressure-induced superconductivity appears at pressures of over 6 GPa as a state neighboring a charge-ordering (CO) state. A pressure-induced superconductor with a low pressure onset has been investigated by various techniques irrespective of 3d transition metal oxides, organic conducting systems and 4f heavy fermion systems. The current borderline lies around a pressure of 2.7–3.0 GPa, because the maximum pressure of a piston–cylinder cell was limited to 2.7–3.0 GPa at most, even if a hybrid WC cylinder was used [6].

Recently, NiCrAl and MP35N cylinders have attracted a lot of attention, because these materials are nonmagnetic and have large tensile and yield strengths [7, 8]. With the development of a NiCrAl pressure cell, the microscopic properties of the superconductivity of $Sr_{14-x}Ca_xCu_{24}O_{41}$ were investigated for the first time [9]. The cuprate spin-ladder system is an example which demonstrates the significance of the NiCrAl pressure cell. The upper limit for a NiCrAl pressure cell is an open problem at present, because the limit strongly depends on the design of the pressure cell as well as the strength of the material.

In the present work, we have applied loads for a piston–cylinder-type NiCrAl pressure cell up to 15 ton, and investigated the pressure limit and efficiency.

2. Experimental conditions

2.1. The pressure cell

We used a NiCrAl cylinder with inner and outer diameters of 6 and 16 mm, respectively. The length of the cylinder was 30 mm. The cylinder was covered by a beryllium–copper (CuBe) sleeve with an outer diameter of 40 mm. A mixture of Fluorinert FC-70 and FC-77 was prepared as a pressure-mediation liquid. A Teflon tube with an inner volume of 4.4 mm diameter $\times$ 15 mm length was filled with the Fluorinert mixture. Both ends were closed by a WC piston and a CuBe plug. The liquid was sealed with CuBe rings set on them. An optical fiber and a NQR coil were used together in the sample space. We used powder samples of Cu_2O with a volume of 2.5 mm diameter and 2.5 mm length, and a coil of 2 mm length with a winding of 25 turns. Ruby powder was fixed using a quick-drying glue on the end of a fiber.

2.2. NQR and ruby fluorescence

NQR and ruby fluorescence are very useful as pressure calibration means because they do not require any sweeps of physical parameters. One of the popular calibration methods at room temperature is Bi resistivity measurement; the other method used at low temperatures is Pb or Sn resistivity measurement. However, the former and the latter require pressure and temperature sweeps, respectively.

Ruby fluorescence is often adopted for diamond anvil cells. The shift of the R1 transition shows a linear relation with pressure. Ruby fluorescence has been confirmed to be a reliable calibration method and the calibration curves have been presented by several studies [10, 11]. The pressure range of our measurements is rather close to that of Piermarni *et al* [10]. They measured pressure up to 19.5 GPa and obtained the fitting curve as

$$P\ (\text{GPa}) = \Delta\lambda\ (\text{nm})/0.365 \qquad (1)$$

where $\Delta\lambda$ represents the shift of the R1 line. In a conventional method using a diamond anvil cell, some pieces of ruby are inserted into the cell. In our measurements with a piston–cylinder-type cell, they were fixed at the end of an optical fiber and inserted with a NQR coil.

^{63}Cu NQR of Cu_2O is also adopted to calibrate pressure because the NQR frequency shows a linear relation with applied pressure. The NQR measurement below 2GPa was carried out at temperatures from 4 to 300 K [12]. The NQR frequency is proportional to the electric field gradient (EFG) at a nucleus site. If the EFG is determined by the surrounding ions, the EFG is

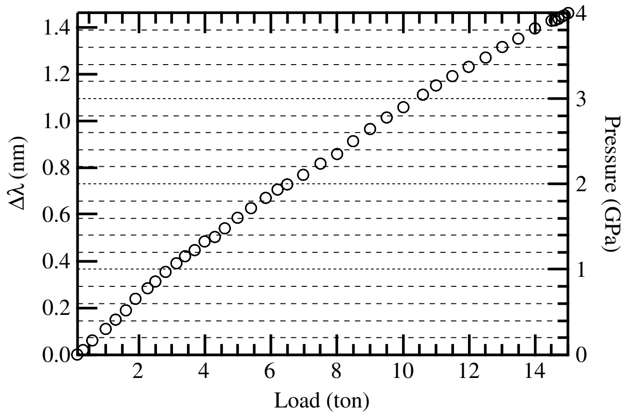

Figure 1. Load dependence of the R1 shift of ruby fluorescence at room temperature. The vertical axis on the right is estimated from equation (1).

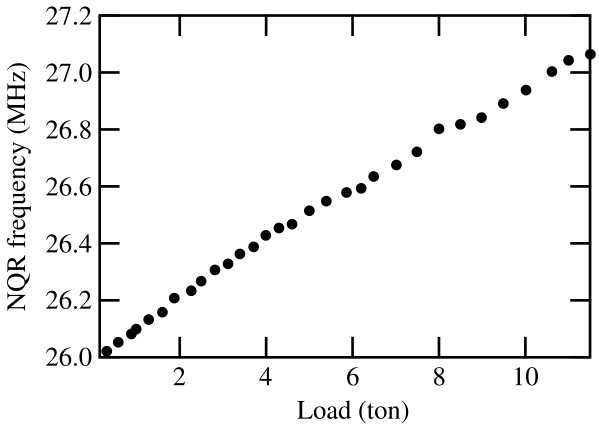

Figure 2. Load dependence of the NQR frequency in Cu_2O. The measurement was performed at room temperature.

proportional to the inverse of the volume. The linear relation implies that the compressibility of Cu_2O is pressure independent. The NQR frequency of Cu_2O is estimated to be

$$\nu \text{ (MHz)} = 26.0 + 0.33P \text{ (GPa)} \quad (2)$$

at pressures below 2 GPa [12, 13].

3. Experimental results

We applied pressure up to 15.0 ton at room temperature using a conventional press, and then effected a return to the initial state. The R1 line moved by 1.47 nm from 694.7 nm on applying a load up to 15.0 ton, as shown in figure 1. Using equation (1), the pressure is estimated to be 4.0 GPa for 15.0 ton. Even at 4.0 GPa no fatal damage was observed for the pressure cell and it was found to be reusable. As seen from the figure, the R1 shift, $\Delta\lambda$, shows a linear response only for a light load. The curve tends to bend downward with an increasing load. The trend becomes remarkable when a heavy load is imposed. The bend is attributed to the

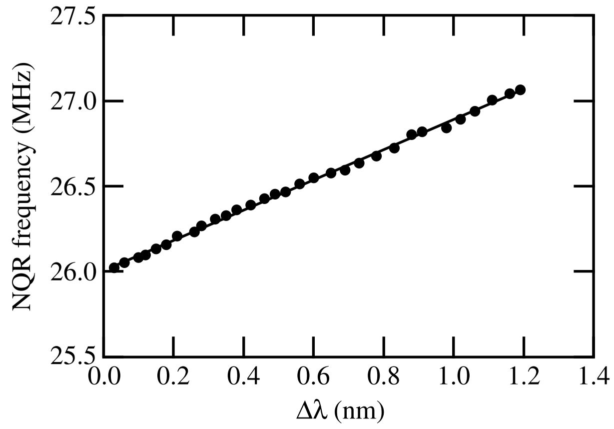

Figure 3. Relation between the NQR frequency of Cu_2O and the shift of the R1 line.

loss of pressure efficiency. The NQR signal also shifted from 26.0 MHz linearly with the slope of 0.93 MHz ton^{-1} at light load, as shown in figure 2. The NQR frequency holds to a linear relation fairly well at light loads, although the linear response breaks down at heavy loads just like that of $\Delta\lambda$.

4. Discussion

The relationship between the NQR frequency and the R1 shift is shown in figure 3. The linearity of the relationship between the two quantities is fairly good. The linear relation is expressed as

$$\nu \text{ (MHz)} = 26.0 + 0.883\Delta\lambda \text{ (nm)}. \tag{3}$$

From equations (1) and (3), we obtained the pressure dependence of ν (MHz) as

$$\nu \text{ (MHz)} = 26.0 + 0.32P \text{ (GPa)}. \tag{4}$$

The relation obtained from our results is consistent with that obtained at pressures below 2 GPa [12].

The pressure efficiency is defined from the results for the R1 shift as

$$\text{efficiency}(\%) \equiv \frac{\Delta\lambda}{0.365}\frac{S}{w} \times 100 \tag{5}$$

where the units of $\Delta\lambda$, the load (w) and the area of the piston (S) are nm, cm^2 and ton, respectively. The efficiency decreases gradually from 95% to 75% during the process of pressurizing from 4 to 15 ton. We found that the inner diameter partly expanded by over 2% after releasing the load.

The loss of efficiency mainly arises from the modification of the CuBe seal ring. The modification becomes remarkable when the ring comes across the expanded region during the pressurizing process. The invention of an effective sealing technique would be a key to raising the efficiency. The critical value of collapse for high strength materials is usually less than 2% or 3%; therefore the upper pressure limit of the NiCrAl cylinder would be at most 4 GPa. In fact, a crack was observed in the WC piston after applying pressure up to 4.0 GPa and releasing pressure down to ambient pressure. The compression strength is 4.5 GPa for nonmagnetic WC, so the appearance of the crack is not so surprising. The conditions that we have imposed seem to be rather close to the limit for the pressure cell.

5. Conclusion

A pressure of 4.0 GPa was achieved using a NiCrAl pressure cell without fatal damage. The relation between the NQR shift of Cu_2O and the R1 shift of the ruby fluorescence was obtained by measuring them simultaneously.

Acknowledgments

We wish to thank Mr D Teshima and Mr T Takahashi for experimental support. The work was partially supported by a Grant-in-Aid from the Ministry of Education, Science and Culture, Japan.

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