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A uniaxial pressure cell for neutron diffraction investigation and its use in studying the single-crystalline $Sr_3Ru_2O_7$ compound

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Abstract
The design and fundamental properties of a uniaxial pressure cell for use with a four-circle neutron diffractometer are reported. It is aimed at facilitating studies on the physical and/or structural properties of solids. The results obtained for a uniaxial-pressure-induced ferromagnet $Sr_3Ru_2O_7$ showed that the unit cell volume increases upon applying uniaxial pressure along the *c*-axis.

Uniaxial pressure (UP) has recently been seen as the key parameter for soft molecular materials and also for systems controlled by the Fermi surface accompanying the spin density wave and the charge density wave, and so on. Among these materials, the bilayered perovskite $Sr_3Ru_2O_7$ shows a crossover from paramagnetism (PM) to ferromagnetism (FM) under not only UP [1, 2] but also magnetic field [3]. The interaction between the rotation of RuO_6 octahedra around the *c*-axis and the FM, which is often discussed in connection with FM correlations in the spin-triplet superconductor Sr_2RuO_4 [4] and the FM in $SrRuO_3$ [5], has attracted much attention in recent years. At ambient pressure, $Sr_3Ru_2O_7$ crystallizes in the orthorhombic *Bbcb* structure, as was reported by Shaked *et al* from powder neutron diffraction studies [6] and confirmed by a single-crystal neutron diffraction experiment combined with convergent-beam electron diffraction (CBED) by Kiyanagi *et al* [7]. To clarify the above magnetism, we have developed a UP cell for neutron diffraction use. In this paper we report its design and fundamental properties and illustrate its application using single crystals of the UP-induced ferromagnet $Sr_3Ru_2O_7$, as an example.

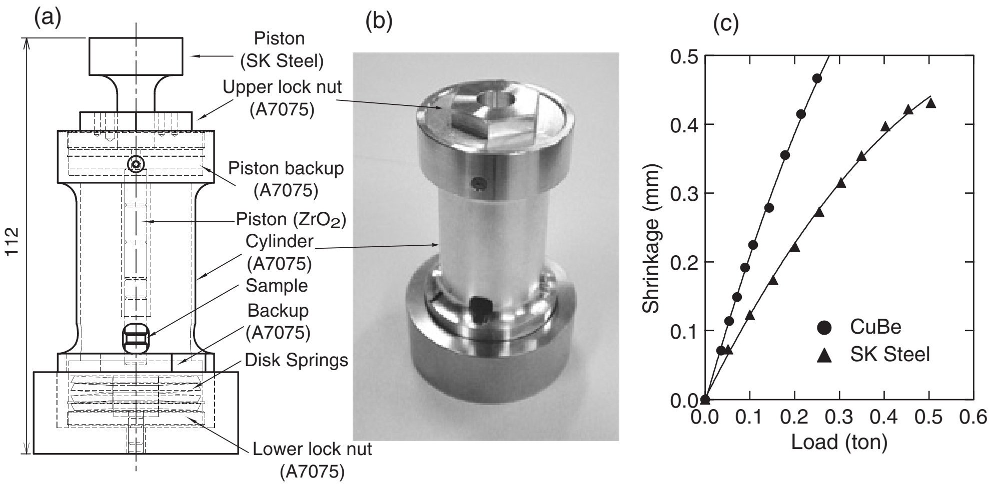

Figure 1. (a) A schematic drawing of the uniaxial pressure cell and (b) a photograph of it. (c) Shrinkage of the cell versus applied forces at room temperature.

The cell was designed for use with the existing 10 K closed cycle refrigerator of a four-circle neutron diffractometer FONDER in the research reactor JRR-3M, JAERI [8]. As shown in figures 1(a) and (b), the aluminium alloy A7075 was used for most of the cell; it has the strongest mechanical properties among aluminium alloys with a small neutron absorption coefficient. The thin cylinder was designed with a large angular aperture to include as many Bragg points as possible. The pistons are made of ZrO_2, which has also good neutron transmission properties [9]. The two kinds of disc springs are made of SK steel (SK-M35)[6] and non-magnetic CuBe alloy. Figure 1(c) shows the shrinkage of the cell versus applied forces at room temperature, illustrating that the maximum load can be about 0.5 and 0.25 ton for one SK steel and one CuBe disc spring, respectively. We can adjust the maximum load from 0.25 to 2.0 ton by using various combinations of stacks of disc springs. Since roughly linear behaviour is shown, the UP was estimated from the applied load divided by the cross section of the crystals.

Single crystals of $Sr_3Ru_2O_7$ were grown by a floating zone method [1]. The three pieces of crystal used in this work are in the shape of thin square plates, with typical dimensions of 3 mm × 3 mm × 1 mm. The cleavage *c*-plane was polished with No 2000 sandpaper to get highly parallel planes. Four-circle neutron diffraction experiments on $Sr_3Ru_2O_7$ under UP along the *c*-axis ($P_{\parallel c}$) have been performed at the FONDER with the wavelength of 1.240 Å [8]. The lattice parameters and the unit cell volume were determined by refining 22 or 32 Bragg reflections. The error bars are estimated by least squares fitting to these reflections. By using the above UP cell, the crystals can be successfully pressurized up to $P_{\parallel c} \sim 4$ kbar and the lattice parameters change significantly.

Figure 2 shows the temperature dependence of the lattice parameters $\langle a \rangle$ $(=(a+b)/2)$ and c for $Sr_3Ru_2O_7$ under ambient pressure from powder neutron diffraction studies by Shaked *et al* [10] and under $P_{\parallel c}$ at 4 kbar from our single-crystal studies for No Y29C-3. At room temperature, one can see that the lattice parameter c shrinks by about 0.3% and $\langle a \rangle$ expands by about 0.3% under $P_{\parallel c} \sim 4$ kbar. Furthermore, the lattice parameter $\langle a \rangle$ shrinks and c expands with decreasing temperature, which is similar behaviour to that indicated by the

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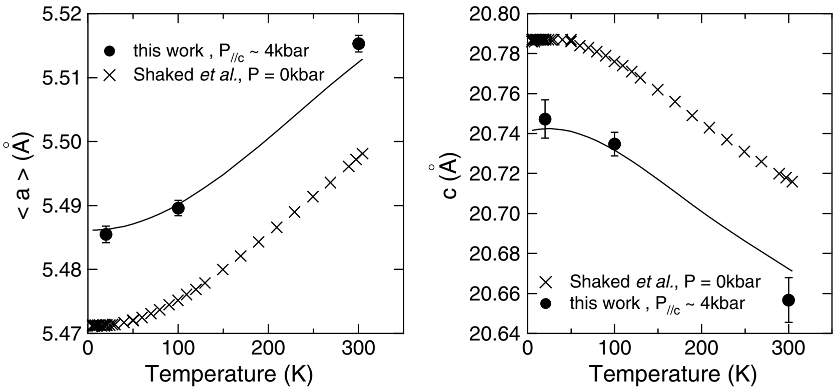

Figure 2. Temperature dependence of the lattice parameters $\langle a \rangle$ $(=(a+b)/2)$ (left) and c (right) in $Sr_3Ru_2O_7$ under $P_{\parallel c}$ (●) and ambient pressure (×) [10]. The solid lines are produced by smoothing the ambient pressure data and shifting them by +0.014 Å and −0.045 Å for $\langle a \rangle$ and c, respectively.

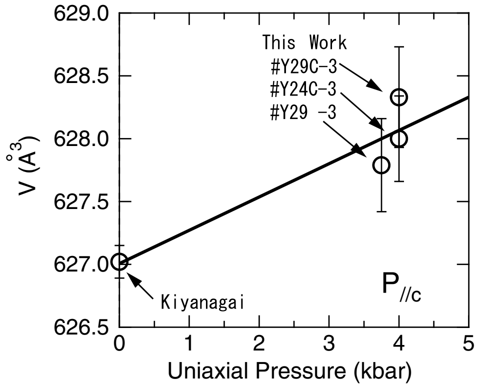

Figure 3. Uniaxial pressure dependence of the unit cell volume V for single-crystalline $Sr_3Ru_2O_7$ at room temperature. The ambient pressure data are from Kiyanagi [7]. The solid line is a guide to the eyes.

ambient pressure data by Shaked *et al* [10] (a guide is given by the solid lines in the figure). Shaked *et al* also reported the existence of a rotation of RuO_6 octahedra around the c-axis and the rotation angle increasing with decreasing temperature. From these findings, it appears that the rotation of RuO_6 octahedra around the c-axis may occur even under $P_{\parallel c} \sim 4$ kbar. But it is unclear whether the rotation angle increases upon applying $P_{\parallel c}$. We need four-circle neutron diffraction studies to reveal whether the UP-induced ferromagnetism is caused by the rotation of the RuO_6 octahedra.

In figure 3 we show the UP dependence of the unit cell volume V for $Sr_3Ru_2O_7$ for three different single crystals, No Y29C-3, No 24C-3 and No Y29-3, together with the single-crystal data for ambient pressure obtained at the FONDER [7]. Very surprisingly, we found the characteristic feature of volume increase for all the crystals. It increases by $0.165 \pm 0.125\%$ under $P_{\parallel c} \sim 4$ kbar, which corresponds to a Poisson ratio exceeding 0.5—as far as we know, there are no materials where it exceeds 0.5 normally. A possible explanation of this unusual feature is the strong correlation of electrons and it may be connected with the rotation of the RuO_6 octahedra.

In summary, we have designed a uniaxial pressure cell for use with a four-circle neutron diffractometer with the maximum load of 2.0 ton. Its application to a uniaxial-pressure-induced ferromagnet $Sr_3Ru_2O_7$ indicated that the crystals do not break up to $P_{\parallel c} \sim 4$ kbar and the unit cell volume increases with $P_{\parallel c}$. In an effort to understand whether the UP-induced ferromagnetism is caused by the rotation of RuO_6 octahedra, four-circle neutron diffraction studies on $Sr_3Ru_2O_7$ under UP are now under way.

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