

実験報告書様式(一般利用課題・成果公開利用)

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	承認日 Date of Approval 2015/06/08 承認者 Approver Ohhara Takashi 提出日 Date of Report 2015/06/08
課題番号 Project No. 2014B0090 実験課題名 Title of experiment Magnetic Structure Analysis of an Antiferromagnet CeNiGe ₃ 実験責任者名 Name of principal investigator Yoichi Ikeda 所属 Affiliation Neutron Science Laboratory, ISSP, The University of Tokyo	装置責任者 Name of responsible person Takashi Ohhara 装置名 Name of Instrument/(BL No.) SENJU (BL18) 実施日 Date of Experiment 09-11 Apr 2015

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
 Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form.
<p>Sample: CeNiGe₃ (single crystal)</p> <p>Mass: 4.6 mg</p> <p>Dimension: ~ 1*1*1 mm³</p> <p>Lattice constants: $a = 21.808 \text{ \AA}$, $b = 4.1351 \text{ \AA}$, $c = 4.1684 \text{ \AA}$ $a^* = 0.288 \text{ \AA}^{-1}$, $b^* = 1.519 \text{ \AA}^{-1}$, $c^* = 1.507 \text{ \AA}^{-1}$</p> <p>Space group: <i>Cmmm</i>, #65</p> <p>Density: 7.319 g cm⁻³</p> <p>Chemical weight: 416.73 g mol⁻¹</p> <p>Neel temperature: $T_N = 5.0 \text{ K}$</p> <p>The single crystalline sample with the dimension of 1*1*1 mm³ was grown by the Ni-Ge binary self-flux method, and was mounted on a thin-Al wire (phi 1mm, 4N) with GE-varnish, as shown in figure 1. The orientation of the principal crystal axes were checked by the high-energy transmission Laue diffractometer installed on ISSP. Typical examples of the Laue photographs are shown in figure 2, where the incident X-ray was parallel to the <i>a</i>-axis for the panel (a) and the <i>c</i>-axis for the panel (b). In addition, the crystal axes and sample quality were cross-checked by the magnetization measurement. As seen in figure 3, the magnetic anisotropy and the metamagnetic fields for the easy-axis are fully consistent with the results of previous report by Mun et al. The fixed sample was installed on the CCR refrigerator with a special low-temperature two-circle Gonio meter on SENJU.</p>

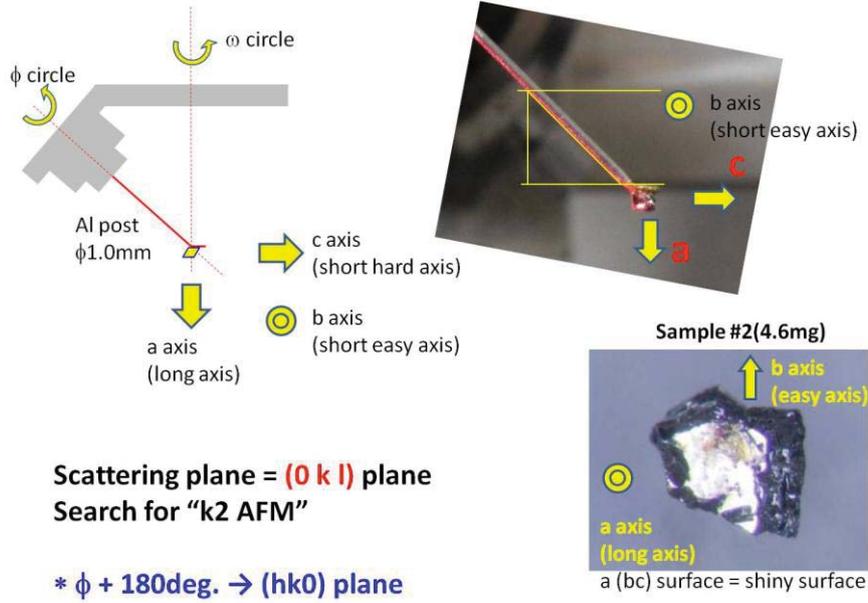


Fig. 1 Experimental set-up. Neutron beam was parallel to the *b*-axis, and the $(0 k l)$ and $(h k 0)$ scattering plane is chosen for searching the incommensurate k_2 -AFM., and the commensurate AFM, respectively.

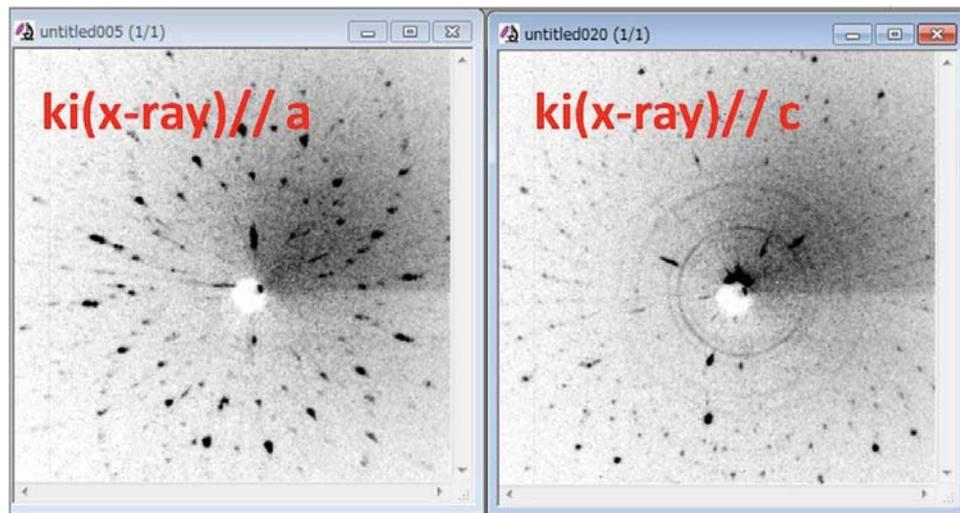


Fig. 2 X-ray Laue photographs of CeNiGe₃. (left) $k_1 // a$ -axis, (right) $k_1 // c$ -axis.

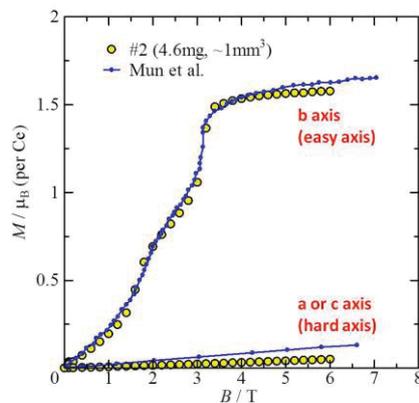


Fig. 3 Magnetization curves of the single crystalline sample of CeNiGe₃ for the magnetically easy-axis (= *b*) and hard axis (*a* and *c*).

2. 実験方法及び結果（実験がうまくいかなかった場合、その理由を記述してください。）

Experimental method and results. If you failed to conduct experiment as planned, please describe reasons.

[Experimental procedure]

CeNiGe₃ is a pressure-induced heavy-electron superconductor and shows the antiferromagnetic ordering below $T_N = 5$ K at ambient pressure. Curiously, the magnetic reflections were observed at two inequivalent positions, i.e. the incommensurate $k_2 = (0, 0.41, 1/2)$ and the commensurate $k_1 = (1, 0, 0)$ positions, in the previous neutron-scattering study with the polycrystalline sample. To clarify this anomalous behavior in CeNiGe₃ at ambient pressure, we searched magnetic reflections in the $(0\ k\ l)$ and $(h\ k\ 0)$ planes with a single crystal sample. First, in order to search the incommensurate magnetic reflections with the propagation vector of $k_2 = (0, 0.41, 1/2)$, the incident neutron beam was parallel to the b -axis where the c -axis was settled in the equator plane, namely the scattering plane is the $(0\ k\ l)$ plane. Next, we searched the commensurate magnetic reflections with the propagation vector of $k_1 = (1, 0, 0)$; here the sample was rotated 180 deg. about the ϕ -axis, and the scattering plane was changed into the $(h\ k\ 0)$ plane. Details of the experiment are shown in Fig. 4.

2015		data acquisition			gonio controller			temperature controller			chopper	
date	run#	begin	end	DAQ time	phi	w	scat. plane	T_CH	T_samp	remarks	frame	remarks
09 Apr.	5281	21:06	23:47	2:41	230	258.75	(0kl)	1.66	3.46 < TN		1st	search for incomm. peaks
09 Apr.	5282	23:47	2:30	2:43	230	264.25	(0kl)	1.66	3.46 < TN		1st	search for incomm. peaks, to cover det. gap
10 Apr.	5283	2:31	6:40	4:09	55	197.5	(hk0)	1.66	3.46 < TN		1st	search for comm. peaks
10 Apr.	5284	6:40	9:23	2:43	55	192	(hk0)	1.66	3.46 < TN		1st	search for comm. peaks, to cover det. gap
10 Apr.	5285	10:17	14:50	4:33	230	258.75	(0kl)	5.49	6.18 > TN		1st	background measurement
10 Apr.	5286	14:55	19:00	4:05	55	197.5	(hk0)	5.49	6.18 > TN		1st	background measurement
10 Apr.	5287	19:08	1:27	6:19	55	197.5	(hk0)	1.67	3.44 < TN		1st	overnight scan for improving statistics
11 Apr.	5288	1:28	6:55	5:27	230	258.75	(0kl)	1.67	3.44 < TN		1st	overnight scan for improving statistics
11 Apr.	5289	6:56	9:01	2:05	55	197.5	(hk0)	1.67	3.44 < TN		1st	to determine UB
11 Apr.	5290	?	?	?	55	199.5	(hk0)	1.67	3.44 < TN		2nd	alignment
11 Apr.	5291	?	?	?	55	200.5	(hk0)	1.67	3.44 < TN		2nd	alignment
11 Apr.	5292	9:50	12:20	2:30	55	200.5	(hk0)	1.67	3.44 < TN		2nd	search for (100) ref.

Fig. 4 History of our experiment.

[Results]

The crystal structure of CeNiGe₃ is the base-centered orthorhombic structure in $Cmmm$ (#64) space group, in which the fundamental Bragg reflections in the $(0\ k\ l)$ plane are allowed at $0kl$ ($k = \text{even}$). This extinction rule for CeNiGe₃ can be clearly recognized in figure 5. In addition, the incommensurate Bragg reflections were observed at $Q = G + (0, \pm 0.41, 1/2)$ where G denotes the fundamental Bragg position. This incommensurate vector is consistent with the previous result of the neutron-scattering study.

Next, we show the contour maps in the $(h\ k\ 0)$ plane to search the commensurate magnetic reflections as observed in the previous study. As seen in figure 6, the fundamental Bragg reflections can be observed for $hk0$ ($h + k = \text{even}$), while there is no Bragg reflections at the forbidden position in the $(h\ k\ 0)$ scattering plane within the experimental accuracy. It is also noteworthy that no significant signal was observed at $(1\ 0\ 0)$ position at which the most intense signal should be observed if the commensurate magnetic ordering is realized below T_N . These results indicate that the commensurate antiferromagnetic ordering is not realized below T_N (at least above 3.46 K) in our single crystal sample. From these results, we conclude that the antiferromagnetic structure of CeNiGe₃ below T_N in zero-magnetic field at ambient pressure is characterized by the incommensurate propagation vector of $k_2 = (0, 0.41, 1/2)$, while not the commensurate one. Further quantitative analysis is now in progress.

2. 実験方法及び結果(つづき) Experimental method and results (continued)

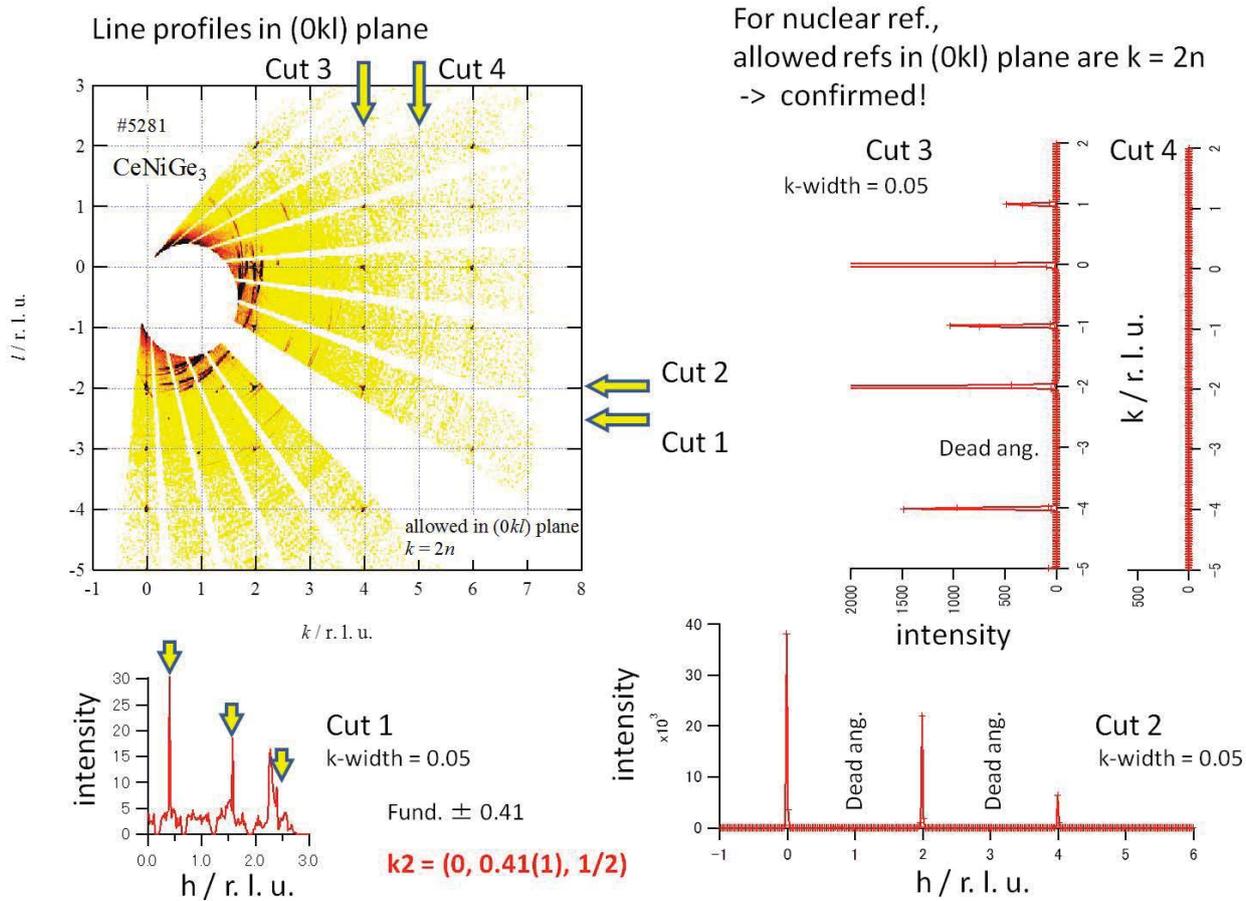


Fig. 5 Contour map and several line profiles in the (0 k l) plane of CeNiGe₃ at 3.46 K (below T_N).

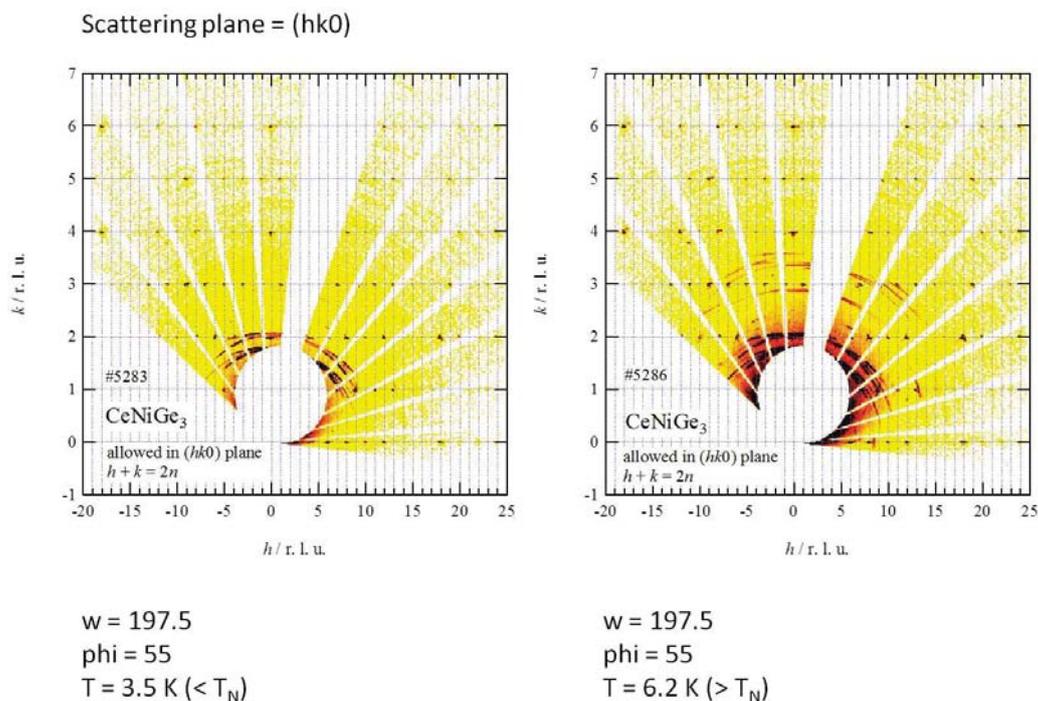


Fig. 6 Contour maps in the (h k 0) plane of CeNiGe₃ at 3.46 K (below T_N) and 6.2 K (above T_N).

Conclusion

We have succeeded to observe the incommensurate magnetic reflections with the propagation vector of $k_2 = (0, 0.41, 1/2)$ in the $(0\ k\ l)$ scattering plane of CeNiGe_3 below T_N . In our single crystal sample, no significant magnetic reflections were observed in the $(h\ k\ 0)$ scattering plane within the experimental accuracy. Accordingly, the antiferromagnetic ordering of CeNiGe_3 in the zero-magnetic field at ambient pressure is characterized by the incommensurate propagation vector of $k_2 = (0, 0.41, 1/2)$, while not the commensurate vector. It is also meaningful that we succeeded to observe the small magnetic signals in a wide k -space with a small amount of the single crystal sample (~ 4.6 mg). After that, we would like to plan the next experiment in high-magnetic fields and under high pressures for revealing the problem in the relationship between the magnetism and the superconductivity.

Future plans

As a future plane, it is interesting to measure the temperature dependence of the propagation vector for revealing the origin of the incommensurate magnetic structure. For this experiment, a ^3He or dilution refrigerator is needed. Furthermore, it is also important to reveal the magnetic structure of the field-induced and pressure-induced magnetic phases for further understanding of the relationship between the antiferromagnetism and the superconductivity in this material. Especially, it is essential to clarify the difference between AFM-I and AFM-II phases in CeNiGe_3 , where the former AFM phase can only coexist with the pressure-induced superconductivity (SC-I). For these experiments, the 7T-class superconducting magnet and the 5GPa-class high-pressure apparatus are required.