High-field, high-pressure, and low-temperature magneto-optical apparatus using a diamond anvil cell

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An optical system equipped with a diamond anvil cell is constructed for studying the optical properties of materials under the simultaneous conditions of high magnetic field, high pressure, and low temperature. As an application of this apparatus, the magnetophotoluminescence due to the exciton state in $Cd_{0.9}Mn_{0.1}Se$ is measured at 4.2 K under 1.25 GPa in the presence of magnetic fields up to 23 T.

I. INTRODUCTION

Pressure modifies the crystal lattice of a material, while the magnetic field gives an additional perturbation to the material through quantization of the electronic states. Therefore, the combination of high pressure and high magnetic field would induce a variety of novel properties of solids. This subject motivates to develop the magneto-optical apparatus by using a diamond anvil cell (DAC) for generating high pressure under high magnetic fields. Recently, by applying the fiber-optic technique, Kuroda *et al.*[1–3] have constructed a cryogenic DAC system which is placed in a Bitter magnet to measure the photoluminescence spectrum of *R* lines in alexandrite at 77 K under the simultaneous conditions of pressure and magnetic field up to 8 GPa and 15 T, respectively. Yamamoto *et al.*,[4] on the other hand, have made a DAC out of a ceramic material, and succeeded in measuring the *R* lines in ruby at 77 K under the pressure up to 7 GPa and the pulsed magnetic field up to 33 T.

In these works, the transmission geometry is employed commonly for detecting the luminescent light. Namely, while a surface of the sample is irradiated by a laser beam, the luminescent light is observed from the back surface of the sample. As far as optically transparent materials such as alexandrite and ruby are concerned, this geometry is convenient for constructing the optical system in a limited bore space of the magnet. However, for the near-edge luminescence in semiconductors, the transmission geometry is inappropriate because the observed spectrum will be affected strongly by the reabsorption due to the interband electronic transition. The same is true for the optical studies of surface states in semiconductors and metals. For such studies, therefore, it is essential to employ the reflection geometry, in which the light flux emitted backward from the surface of the sample is detected.

In this article, we describe a reflection-type system equipped with a miniature DAC. The system is designed to fit a hybrid magnet which can generate steady magnetic fields up to 28 T in its bore of 52 mm.[5] Nowadays, a superconducting magnet which can generate 20 T in 52-mm bore is commercially available. Our apparatus is adaptable also to such a superconducting magnet. As an example of the experimental result, new data of the magnetoluminescence due to the recombination of excitons in $Cd_{0.9}Mn_{0.1}Se$ at 4.2 K are presented.

II. DESIGN AND CONSTRUCTION

There have been a few reports of the apparatus for the luminescence measurement, which employs a single line of optical fiber in the reflection geometry.[6,7] The optical fiber of this system plays a dual role, that is, the guide of the light source as well as the collector of the luminescence light. It is known that in this single-fiber method the fluorescence of the fiber itself superposes the signal from the sample.[8,9] This problem is particularly serious for the experiment with a strong-field magnet where a long fiber cable has to be used for safety reasons as well as for reducing the leakage flux of the magnet. For the present purpose, therefore, we have to employ two independent fibers, one introducing the light source and the other collecting only the luminescent light.

Figure 1 shows the experimental setup for the low-temperature magnetoluminescence spectroscopy using our apparatus. A metal Dewar of inner diameter 44 mm is installed in the hybrid magnet. Hence the outer diameter of the optical chamber is limited to 42 mm. The two optical fibers are fed into the chamber through the vacuum fittings with O rings.[6]

Figure 2 shows the arrangement of DAC and optics inside the optical chamber. A metal-gasketed miniature DAC is used to generate a high pressure. This DAC is a modification of a Merrill–Bassett cell.[10] The anvil-support plates are fastened by three screws, and clamped by three other screws. The diamond rocker has a conical slot at its center with an aperture angle of 60°. This DAC is designed to have an outer diameter 30 mm in order to meet the spatial requirement mentioned above. As has been pointed out by Dunstan and Scherrer,[11] the small size of the DAC does not necessarily cause disadvantage in generating a high pressure. In fact, our DAC can generate pressure up to 10 GPa as easily as a 42-mm-diam DAC[1–3] of the same type.

The light source is introduced directly into the optical chamber through a cable (Showa Electric Wire & Cable, CF100/140) of glass fiber of core diameter 100 μm and numerical aperture 0.28. The fiber is covered by a PVC tubing of outer diameter 3 mm. At its end, the PVC cover

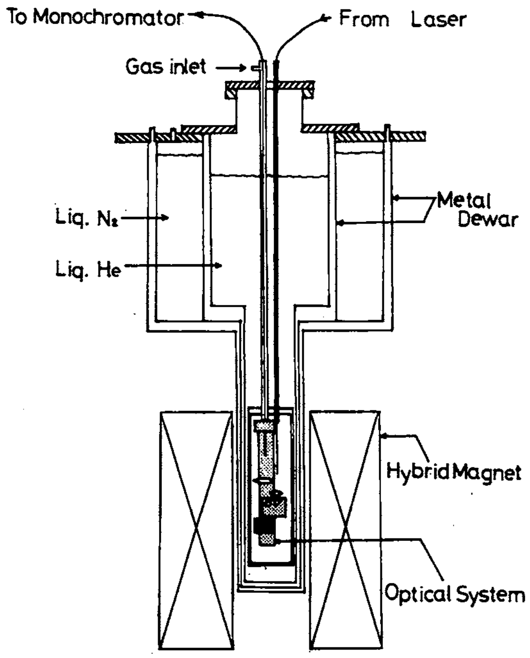

FIG. 1. Experimental setup for low-temperature magneto-optical spectroscopy under high pressures in high fields.

is replaced by a 50-mm-long and 3.2-mm-diam sleeve of stainless steel. This end is inserted in its holder and settled by screws as shown in Fig. 2. The inner diameter of the holder is 6 mm, so that the position of the fiber can be adjusted by the screws. The light source is focused on the sample in the DAC by using a lens and two prisms. Since the sample is as small as about $100\times100\ \mu m^2$, the alignment of the light source, lens, and prisms should be made accurately. Figure 3 illustrates the mechanism for adjusting the vertical position of the lens and the horizontal positions and angles of the prisms. A good alignment can be obtained easily by making the adjustments to the optics

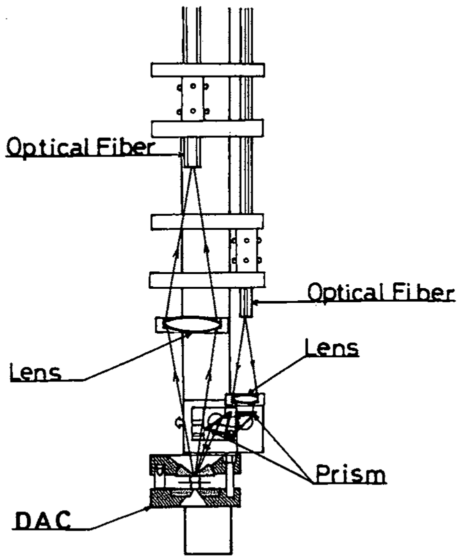

FIG. 2. Arrangement of a miniature DAC and optics.

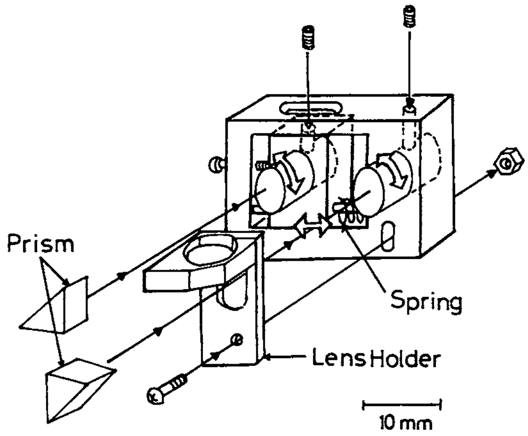

FIG. 3. Mechanism for aligning optics and light source.

while looking at the shadow of the sample which is shed from the outer side of the DAC.

The light emitted backward from the sample is collected by another lens; it is fed into a 1.48-m-long silica fiber (Mitsubishi Cable Industries, ST1000H-LG), as shown in Fig. 2. This fiber has a diameter of 1 mm, and has a sleeve made of a stainless-steel tube of outer diameter 4 mm. The position of its end can be adjusted by screws so as to catch the image of the sample. Note that in the present geometry the flux of the light source reflected by the table of the diamond goes outside the aperture of the light-collecting lens. If the surface of the sample is flat, the reflected light flux is also mostly lost. Consequently, the present apparatus is basically free from the fluorescence of the optical fiber. Nevertheless, it is impossible to separate the flux of the light source completely from the luminescent light because there is a random reflection due to the roughness of the surface of the sample. Therefore, if the fluorescence of the fiber-cable linking between this cryostat and the spectrometer is not negligible compared with the signal level, an optical filter which cuts the light source is inserted between the light-collecting lens and the silica fiber.

The optics holders, the base plate, and the cap of the chamber are all made of brass. The collected light is transmitted to the spectrometer through a cable of bundled silica fibers.

III. OPERATION

To test the operation of the present system, we measure the luminescence spectrum due to the recombination of photocreated excitons in a diluted magnetic semiconductor $Cd_{0.9}Mn_{0.1}Se$. The diamonds used as anvils have a culet 0.6-mm-diameter, a girdle 3.2-mm-diameter and a table 2.0-mm-diameter. The gasket is a stainless-steel plate of thickness 0.2 mm. Pressure is generated in the 0.3-mm-diam hole of the gasket. A 4:1 mixture of methanol and ethanol is used as the pressure medium. Pressure is calibrated by the shift of R lines of a chip of ruby. An Ar ion laser is used as the light source for exciting both the sample

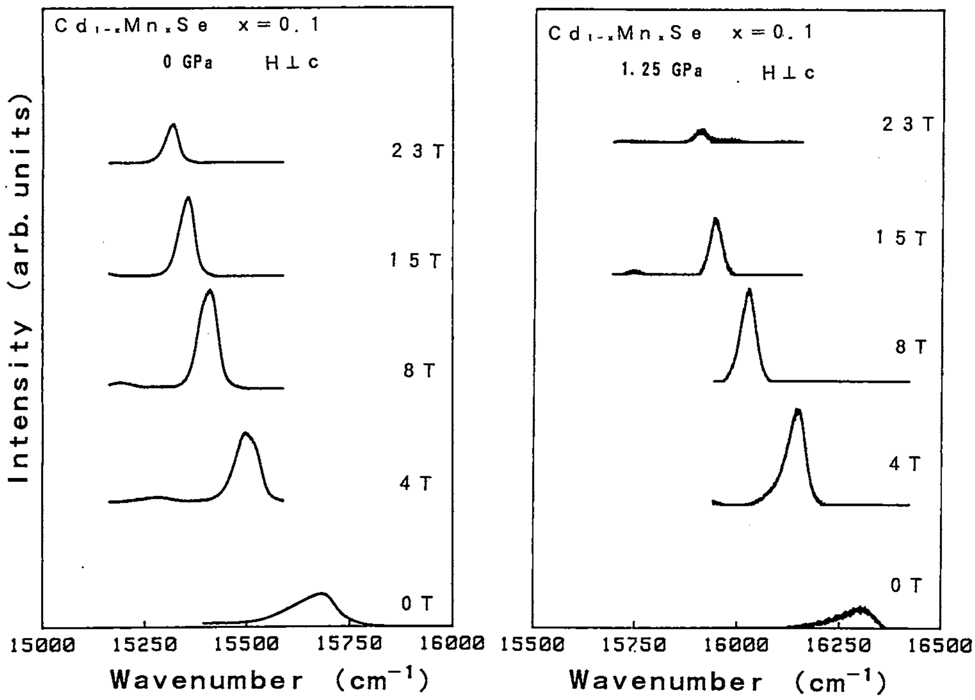

FIG. 4. Magnetoluminescence spectra in $Cd_{0.9}Mn_{0.1}Se$ at 4.2 K under 1 atm (left) and 1.25 GPa (right). Magnetic field is applied normal to the *c* axis of the crystal. The 514.5-nm line of an Ar-ion laser is used as the light source.

and ruby. The optical chamber is filled with helium gas, and is immersed in liquid helium at 4.2 K.

Figure 4 shows the photoluminescence spectra observed at 1 atm and 1.25 GPa under several magnetic fields, where the field is applied normal to the *c* axis of the wurtzite structure of the $Cd_{0.9}Mn_{0.1}Se$ crystal. Note that the pressure causes a blue shift of the spectrum at zero magnetic field. At both pressures, quite a large Zeeman shift which is characteristic of diluted magnetic semiconductors at a low temperature[12] is clearly observed as the field increases up to 23 T, the maximum field examined in the present work.

No serious problem has happened with the apparatus even under these extreme conditions. In particular, although the glass-fiber cable CF100/140 is not manufactured originally for the cryogenic use, our experiment shows that the temperature-cycling between room temperature and 4.2 K can be repeated many times.

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[1] Y. Oeda, M. Takeda, N. Kuroda, G. Kido, Y. Nishina, and Y. Nakagawa, *Advances in Magneto-Optics, Proceedings of the International Symposium on Magneto-Optics*, J. Magn. Soc. Jpn. Vol. 11, Suppl. S1, edited by K. Tsushima and K. Shinagawa (The Magnetic Society of Japan, Tokyo, 1987), p. 157.
[2] N. Kuroda, Y. Oeda, G. Kido, M. Takeda, Y. Nishina, and Y. Nakagawa, High Press. Res. **2**, 65 (1989).
[3] N. Kuroda, H. Kanda, G. Kido, M. Takeda, Y. Nishina, and Y. Nakagawa, Jpn. J. Appl. Phys. **31**, L700 (1992).
[4] K. Yamamoto, S. Endo, Y. Yamagishi, H. Mikami, H. Hori, and M. Date, Rev. Sci. Instrum. **62**, 2988 (1991).
[5] Y. Nakagawa, K. Noto, A. Hoshi, K. Watanabe, S. Miura, G. Kido, and Y. Muto, Physica B **155**, 69 (1989).
[6] D. Heiman, Rev. Sci. Instrum. **56**, 684 (1985).
[7] H. Ackermann, E. D. Jones, J. E. Schirber, and D. L. Overmyer, Cryogenics **25**, 496 (1985).
[8] E. D. Isaacs and D. Heiman, Rev. Sci. Instrum. **58**, 1672 (1987).
[9] D. Heiman, X. L. Zheng, S. Sprunt, B. B. Goldberg, and E. D. Isaacs, *Proc. SPIE-Int. Soc. Opt. Eng.*, edited by F. Adar, J. E. Griffiths, and J. M. Lerner (SPIE, Bellingham, WA, 1989), Vol. 1055, p. 96.
[10] A. Jayaraman, Rev. Sci. Instrum. **57**, 1013 (1986).
[11] D. J. Dunstan and W. Scherrer, Rev. Sci. Instrum. **59**, 627 (1988).
[12] A. J. Gaj, *Diluted Magnetic Semiconductors, Semiconductors and Semimetals*, edited by J. K. Furdyna and J. Kossut (Academic, New York, 1988), Vol. 25, Chap. 7.