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A.C. SUSCEPTIBILITY MEASUREMENTS UNDER HIGH PRESSURE UP TO 2.0 GPa

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A newly developed pressure setup for a.c. susceptibility measurements in the pressure range up to 2.0 GPa is presented; the temperature domain extends from 77 to 450 K. The steel pressure chamber contains the sample located at the center of a set of three compensated pick-up coils and the pressure and temperature sensors. Either alcohol or extraction naphtha is used as the liquid pressure transmitting medium. The (P,T) magnetic phase diagram of $(Fe_{0.975}Ni_{0.025})_2P$ system in the pressure range up to 2.0 GPa is reported.

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1. Introduction

The aim of this paper is to describe a new method developed for measuring a magnetic a.c. susceptibility under high pressure up to 2.0 GPa. It may be applied to construct (P,T) phase diagrams of magnetic systems. The a.c. magnetic susceptibility under high pressure up to 1.5 GPa was previously measured by several authors (see [1] and references therein). Recently we have presented a new pressure apparatus for magnetization measurements under pressure up to 0.35 GPa and in high magnetic field up to 20 T (produced by a resistive Bitter magnet) [2]. In the two previous cases, a Be–Cu pressure chamber and gaseous helium as a pressure-transmitting medium were used. The pressure chamber may be also realized in Ti–Cu alloy in the magnetic field up to 20 T being produced by a superconducting magnet [3]. Two devices which allow accurate magnetization and susceptibility measurements under high pressure have been reported by Haselwimmer [4] and Ishizuka [5]. In both setups, a diamond anvil cell (DAC) associated to a SQUID magnetometer was used; the maximum pressure attains 50 (15) GPa for the magnetization (susceptibility) measurements respectively but the pressure was not hydrostatic.

To conclude this introduction, it is mentioned that a review of the apparatus for magnetization and susceptibility measurements under high pressure has been recently proposed by Schilling [6].

2. Description of the apparatus

In our high pressure generator manufactured by the Polish firm Unipress (Warsaw, Poland), the quasi-hydrostatic pressure up to 2.0 GPa results from the compression of a liquid in a steel chamber by a piston. A nonreactive liquid, namely liquid alcohol or extractive naphtha was used as the pressure transporting medium.

The pressure chamber contains the main elements of the experiment (Fig. 1):

- a solenoid (named the "field coil") wound on a teflon cylinder creates a sinusoidal magnetic field of weak amplitude at a frequency of some hundred hertz;
- the powder sample is contained in a teflon sample holder located at the center of the central pick up coil;
- the pressure and the temperature sensors consist of a manganin pick-up coil and a copper–constantan thermocouple;
- three pick-up coils wound on a slotted copper tube are connected in series--opposition.

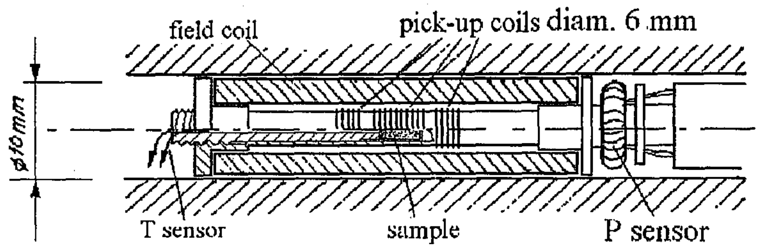

Fig. 1. Pick- up coils for a.c. susceptibility measurements inside the pressure chamber.

One delicate question concerns taking out the thin (0.1 mm) wires of the pressure chamber; the use of a conic feedthrough, securing the continuity of the wire's material appears to be a satisfying solution.

In the presence of a magnetic sample the signal induced in the precompensated pick-up coils is proportional to the magnetic susceptibility and can be measured by means of a selective nanovoltmeter. However, the precision of the measurement is mainly determined by the quality of the compensation. To improve the compensation in the absence of a sample, the magnetic flux induced in two extra small coils is subtracted (or added) through a potentiometer to the main signal delivered by the three pick-up coils.

To get the sample temperature variations, a heater (double spiral) is wound on the external surface of the pressure chamber. First, the cooling of the sample is obtained by plunging the pressure chamber into the liquid nitrogen bath. The temperature changes are then regulated so that a good reproducibility of our experimental results is obtained. The temperature measurement was carried out through the use of the copper–constantan thermocouple placed in a direct contact with the sample. As previously observed [7], the influence of pressure on the accuracy of the temperature measurements is negligible. On the contrary, the influence of temperature on the pressure sensor cannot be neglected. The corrections proposed by Dmowski and Litwin-Staszewska [8] (see also [9]) have then been taken into account to determine the real pressure values. Finally, the pressure measurement accuracy can be estimated to about 3%.

The signal detected in the pick-up coils, the temperature and the pressure values were directly registered by means of a PC. The a.c. susceptibility measurements of the pressure chamber background were carried out without a sample and no significant influence on the magnetic signal of the pressure vessel was detected. In our experiment the value of the signal proportional to the a.c. magnetic susceptibility of the sample was measured. For each pressure value, the variation of the a.c. susceptibility versus temperature was determined (Fig. 2).

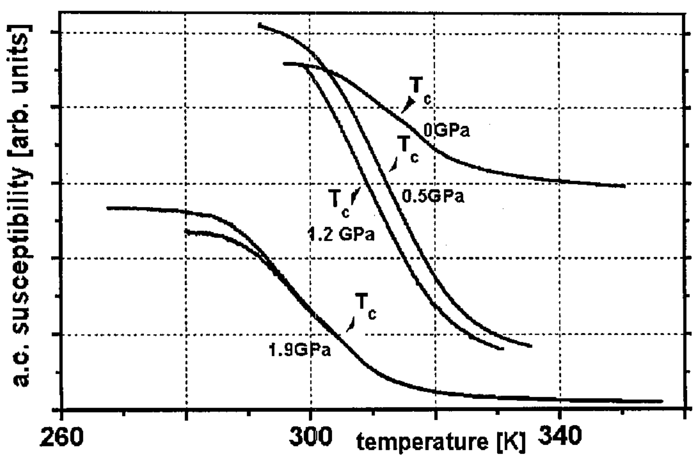

Fig. 2. Temperature variation of the a.c. susceptibility of $(Fe_{0.975}Ni_{0.025})_2P$ at different pressures. The pressure values indicated in the figure correspond to the inflection point of the appropriate curve.

When the signal decreases with increasing temperature, there exists an inflection point. Its abscissa was defined as the Curie temperature T_C. The PC registers the temperature, pressure and a.c. susceptibility with the frequency of 0.2–0.5 Hz during the experiment time. In the vicinity of the inflection point a.c. susceptibility signal after each experimental cycle could be approximated by the polynomial $a_0 + a_1T + a_2T^2 + a_3T^3$. We usually use about 300 points that corresponds to $T_C \pm 15$ K. The temperature of the inflection point is determined as $-a_2/3a_3$ (i.e. extremum of the first derivative of the polynomial used). In these experiments the experimental error of T_C depends on the accuracy of the temperature measurements which are done by means of the copper–constantan thermocouple. It means that the experimental error of T_C is equal to 1 K. For each sample we could repeat this measurement for different pressure values. Based on the pressure dependence of T_C, the (P,T) magnetic phase diagrams were then constructed. An illustration of our apparatus capability is shown in Fig. 3 where the (P,T) diagram

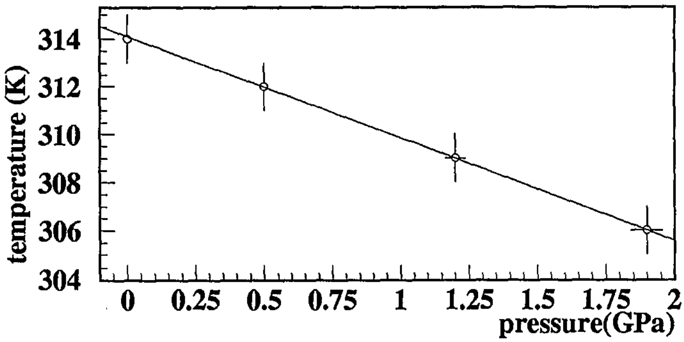

Fig. 3. (P,T) magnetic phase diagram for $(Fe_{0.975}Ni_{0.025})_2P$ at the pressure up to 2 GPa.

of a $(Fe_{1-x}Ni_x)_2P$ sample with $x = 0.025$ is reported. The $(Fe_{1-x}Ni_x)_2P$ system crystallizes within the hexagonal Fe_2P-type structure. For $x < 0.8$ the compounds exhibit ferromagnetic properties. The increase in T_C for $x < 0.1$ and the strong decrease in T_C for $x > 0.1$ was established. Moreover, the pressure dependence of T_C was previously studied by the self-inductance method and by the a.c. susceptibility method up to 0.5 GPa [10] and 1.2 GPa [11], respectively. Our results remain in good agreement with those previously published.

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