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Magnetic properties of the pressure-induced ordering state in $YbInCu_4$ investigated with NMR, magnetization, and x-ray diffraction measurements

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We have investigated the newly discovered pressure-induced magnetic ordering phase in $YbInCu_4$ using dc magnetization, nuclear magnetic resonance (NMR), and x-ray diffraction measurements. The dc magnetization measurements provide evidence for the appearance of spontaneous magnetization above 3 GPa and enable us to establish a pressure-temperature phase diagram for $YbInCu_4$ over a wide pressure range up to 27 GPa. It is shown that, while the valence transition temperature T_V is strongly reduced with increasing pressure, the magnetic ordering temperature T_M reveals a very weak pressure dependence. We find no evidence for any structural phase transition up to 15 GPa within the experimental accuracy. The Curie-Weiss temperature dependence of susceptibility in pressure-stabilized paramagnetic state is evaluated from the Knight shift data obtained from ^{115}In NMR measurements. From these experimental results, we discuss possible electronic states of the $4f$ electrons in the high pressure magnetically ordered phase of $YbInCu_4$.

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I. INTRODUCTION

Since the first synthesis by Felner and Nowik,[1–3] $YbInCu_4$ has attracted much attention, because it undergoes first-order isostructural valence transition that is similar to the α-γ transition in cerium metal.[4] At ambient pressure, both phases above and below the transition temperature T_V=42 K are well characterized by the temperature dependences of susceptibility, i.e., in the high temperature phase, the susceptibility exhibits Curie-Weiss behavior with an effective moment close to the full free-ion moment of Yb^{3+}, $4.54\mu_B$, indicating that $4f$ electrons are in a well-localized state.[2,5] On the other hand, the susceptibility in the low temperature phase is nearly temperature independent (Pauli paramagnetism). This is interpreted as a mixed-valence state with Yb valence of approximately 2.9.[2]

This valence transition strongly depends on hydrostatic pressure.[6–10] By applying pressure of 2.4 GPa, the low temperature phase is suppressed below 2.5 K and a new magnetically ordered phase appears above 2.4 GPa. This finding has renewed interest in this compound.[10] In fact, in a variety of mixed-valence Yb-based compounds in which the electronic configuration is between trivalent $4f^{13}$ and divalent $4f^{14}$ the trivalent state can be stabilized relative to the larger divalent state under high pressure.[11] Attempts to observe pressure-induced nonmagnetic-magnetic transition in Yb-based compounds have been reported for the last few decades.[12] Among the previously reported cases of the pressure-induced magnetic order in Yb-based compounds, the rather low critical pressure of P_C=2.4 GPa and the value of the ordering temperature (T_M=2.4 K) of $YbInCu_4$ allow one to perform precise studies of the physical properties of the system in the vicinity of magnetic and valence transitions. Previous ac susceptibility measurements on $YbInCu_4$ suggest a ferromagnetically ordered state under high pressure.[10] This magnetically ordered state is confirmed at a microscopic level by high pressure nuclear quadrupole resonance (NQR) measurements.[13,14] However, neither direct experimental information about magnetic structure nor a phase diagram over a wide range of pressure has been presented so far. Thus, the prime motivation of this work is to perform a comprehensive investigation of the ground state properties of $YbInCu_4$ under high pressure.

In this paper, we report dc magnetization, nuclear magnetic resonance (NMR), and x-ray diffraction studies on $YbInCu_4$ under high pressure. The dc magnetization measurement over a wide pressure range gives evidence for spontaneous magnetization above 3 GPa. Our constructed pressure-temperature phase diagram up to 27 GPa for $YbInCu_4$ shows that, once magnetic ordering appears above P_C, T_M shows very weak pressure dependence. From the x-ray diffraction measurement, there is no evidence for any structural phase transition up to 15 GPa. In addition, we deduced the temperature dependence of the spin susceptibility in the pressure-stabilized paramagnetic phase from ^{115}In-NMR Knight shift measurements up to 2.6 GPa. The analysis of the data indicates that large effective magnetic moment (close to the free Yb^{3+} ion) is still retained at 2.6 GPa. Finally, on the basis of these experimental results, we discuss possible electronic states for $4f$ electrons in the pressure-stabilized phase.

II. EXPERIMENTAL DETAILS

Single crystals of $YbInCu_4$ were grown using the flux method as described in Ref. 15. For the dc magnetization measurement, a diamond-anvil cell (DAC) was used to search for magnetic properties in a wide range of pressure. The experimental setup consists of a vibrating coil magnetometer (VCM) using a superconducting quantum interference device (SQUID) sensor. The SQUID detection coil is located several hundreds μm away from the gasket. It detects the gradient of the magnetic flux, whose signal intensity is proportional to the magnetization of the sample. At zero field, residual field at the sample position is negligibly small (less than 5 mOe). The details of the experimental setup are described elsewhere.[16,17] The use of the SQUID VCM leads to a significant improvement of the signal-to-noise ratio, and thereby allowed us to measure the dc susceptibility under pressures exceeding 10 GPa using the DAC technique. Both the valence transition (below P_C) and the magnetic transition (above P_C) were detectable. A mixture of 4:1 methanol-ethanol was used as a pressure-transmitting medium. Pressure was monitored *in situ* by a superconducting Pb manometer. Therefore the calibration of pressure for the present experiment is limited to ~27 GPa, above which the superconducting signal of Pb is not observable down to the lowest temperature of ~1.5 K.

In order to perform accurate measurements of the dc susceptibility in the pressure-induced paramagnetic state, we have carried out ^{115}In-NMR measurements under pressure. The Knight shift, which is obtained from the NMR study, is intimately related to the uniform susceptibility through the hyperfine field. The NMR measurements were performed using a phase-coherent spin-echo spectrometer, and the Knight shift was obtained by analyzing the peak position of the NMR spectrum as a function of field around 4 T. Hydrostatic pressure for the NMR measurements was produced by using a piston-cylinder pressure cell made of nonmagnetic NiCrAl/CuBe alloy with polyethylsiloxane as a pressure-transmitting medium.[18] The pressure dependence of the lattice constants at 300 K up to 15 GPa was measured on a powder sample by energy dispersive x-ray diffraction at HASYLAB (beamline F3) using the DAC technique and liquid nitrogen as pressure medium. The magnetization measurements were carried out on a small piece of the single crystal. For the NMR measurements, a powder sample was used, whose grain size was a few hundreds μm or less.

III. RESULTS AND DISCUSSION

The magnetization vs temperature (M-T) curves were obtained by the following processes (i)–(iv):

(i) After zero-field-cooling (ZFC) down to the lowest temperature, the temperature is increased at certain finite field.

(ii) Temperature is lowered down to the lowest temperature in the field.

(iii) Magnetization is measured with zero-field-heating (ZFH).

(iv) Magnetization is measured with ZFC.

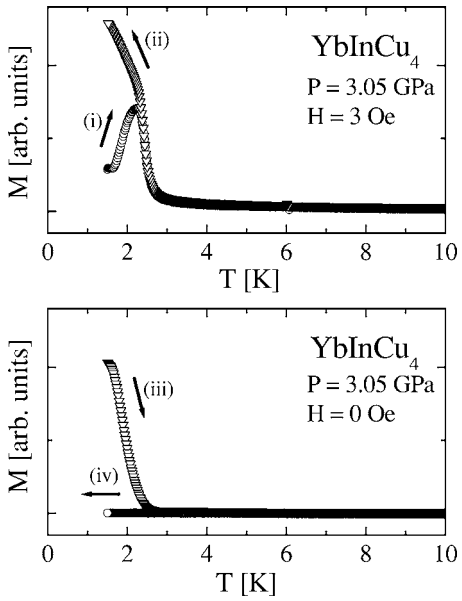

FIG. 1. M-T curves at 3.05 GPa. Upper panel: (i) and (ii) denote the field-heating and field-cooling processes, respectively. Measurements were carried out in a field of 3 Oe after ZFC. Lower panel: M-T curves (iii) and (iv) are ZFH and ZFC processes, respectively, after the processes of (i) and (ii).

In Fig. 1, we show representative M-T curves under pressure. The large evolution of the magnetization observed below 2.5 K in process (iii) gives direct evidence for spontaneous magnetization in the high pressure phase of $YbInCu_4$. In process (iv), this increase in the magnetization could not be seen. This is due to the remarkable hysteresis in the M-T curves between increasing and decreasing temperature cycles, which is characteristic for ferromagnetic ordering.

The temperature dependence of the magnetization at several pressures are shown in Fig. 2, where all the data were taken according to process (iii) after magnetized by 3 Oe. No sign of magnetic ordering was observed at 0.98 GPa, except for steplike temperature dependence originating from the va-

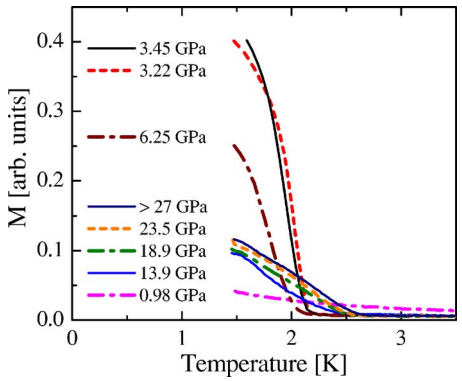

FIG. 2. (Color online) Temperature dependence of magnetization at various pressures of 0.98, 3.22, 3.45, 6.25, 9.25, 13.9, 18.9, 23.5, and >27 GPa. The notation of >27 GPa for the highest pressure implies that the pressure is higher than 27 GPa, because the superconducting signal of Pb manometer was not observable down to the lowest temperature of ~1.5 K.

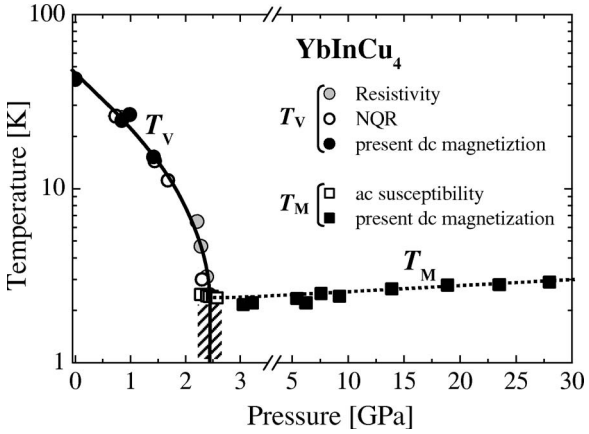

FIG. 3. T-P phase diagram on a semilog scale for $YbInCu_4$. T_M is the magnetic ordering temperature taken from ac χ measurements (Ref. 10) and present results, and T_V is the valence transition temperature from resistivity and NQR data (Refs. 10 and 13) and present measurements. The solid and dotted lines indicate the first- and second-order phase transitions. Note that the mixed-valence and high pressure ordering phases coexist in the hatched area near P_C (Ref. 13).

lence transition (not shown). The resulting pressure dependence of the onset temperatures T_M and T_V are summarized in the pressure-temperature (P-T) phase diagram (Fig. 3), together with previously reported data of T_M and T_V.[10,13] T_V drastically decreases with increasing pressure. On the other hand, once T_M abruptly appears at P_C, T_M shows very weak pressure dependence up to 27 GPa. The hatched area in Fig. 3 indicates that the high pressure magnetically ordered phase coexists with the mixed-valence phase. High pressure NQR experiments reveals that the two phases coexist at least up to 2.6 GPa.[19] These features support that the transition between the two low temperature phases, the mixed-valence and the magnetically ordered phases, is of first order with respect to pressure. This implies that there is no quantum critical point (QCP) in the P-T phase diagram for $YbInCu_4$, as suggested from previous high pressure NQR studies.[13,14] The NQR measurements indicate that the whole phase boundary between the low temperature mixed-valence state and the adjacent phases is of first-order, and the magnetic ordering for $P > P_C$ is of second order.[13,14] These characteristics of transitions are represented in Fig. 3 by the solid (first order) and dotted (second order) lines.

To obtain information about possible pressure-induced structural phase transition that could be related with the sharp transition from the mixed-valence to the magnetically ordered states, we have performed energy dispersive x-ray diffraction on $YbInCu_4$ up to 15 GPa. Figure 4(a) shows some selected x-ray diffraction patterns obtained at room temperature and at different pressures. The estimated pressure dependence of the lattice parameter a is shown in Fig. 4(b). We find systematic shift of the diffraction lines due to volume reduction in the pressure range up to about 15 GPa. Hence the sharp transition from the mixed-valence to magnetically ordered states at $P_C = 2.4$ GPa is not connected with a structural phase transition. We find changes of the intensity of some diffraction lines (220 and 311). Such a change of the

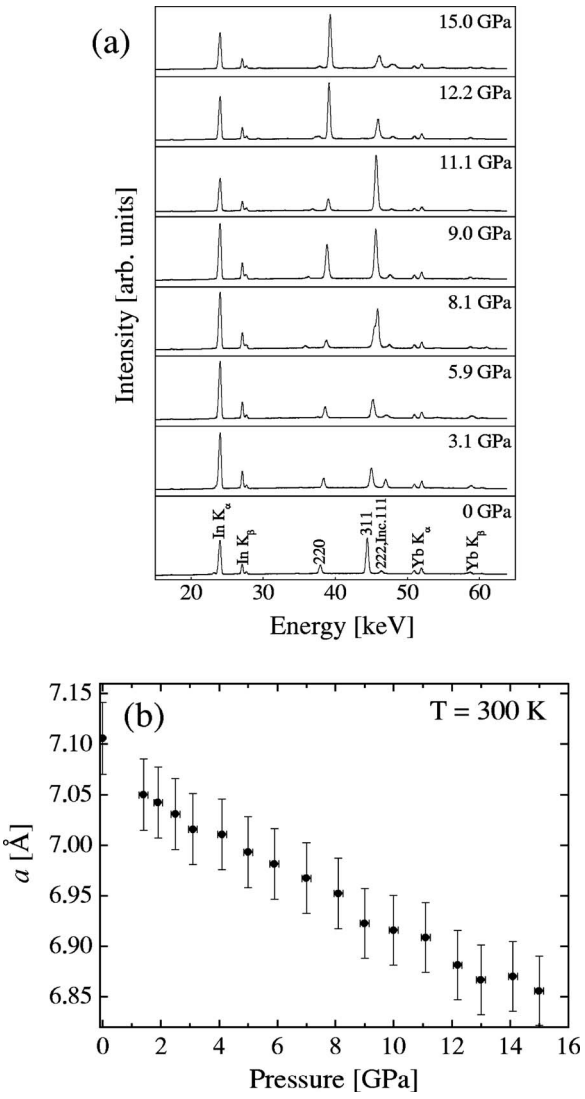

FIG. 4. (a) Room temperature energy dispersive diffraction patterns of $YbInCu_4$ obtained at several pressures up to 15 GPa. (b) Pressure dependence of the lattice parameter a.

relative weight of the line intensity with pressure is obviously related to the texture of the powdered sample. The focused x-ray beam is not exactly hitting the same part of the sample at different pressures. However, this effect has no consequences on the pressure-induced changes in the lattice parameters and the volume. The lattice parameter smoothly decreases with increasing pressure up to 15 GPa as seen in Fig. 4(b) and the pressure-volume relationship was fitted using the Birch equation,[20]

$$P(x) = \tfrac{3}{2}B_0(x^{-7} - x^{-5})\left[1 - \tfrac{3}{4}(B^* - 4)(1 - x^{-2})\right],$$

where $x = (V/V_0)^{1/3}$ is a reduced length. The fit gives a Bulk modulus $B_0 = 102$ GPa and a pressure derivative B^* of about 15. The estimated value of B_0 is in good agreement with that deduced from the temperature dependence of the elastic constants.[21] This value is also close to that for $YbAgCu_4$.[22] Thus, within the accuracy of our experimental data, there is no evidence for any structural phase transition up to 15 GPa.

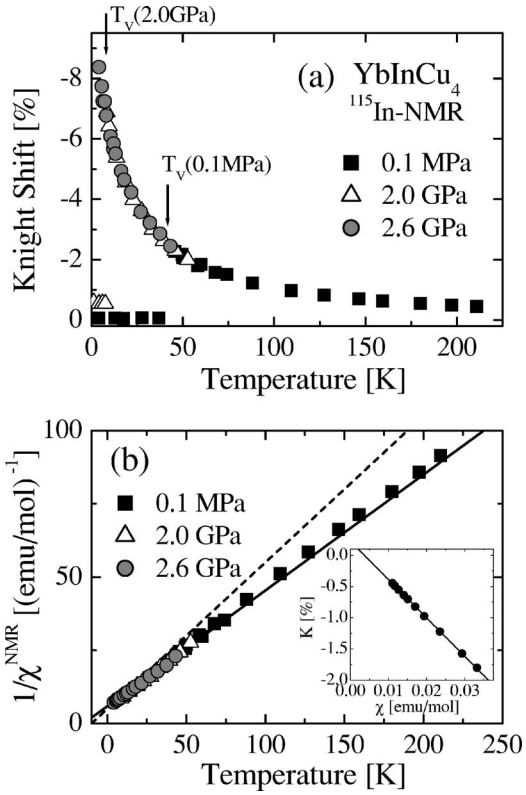

FIG. 5. (a) Temperature dependence of the Knight shift K measured on In-site at ambient pressure, 2.0 GPa and 2.6 GPa. $T_V(P)$ is 42 K (at ambient pressure) and 8 K (at 2.0 GPa). For 2.6 GPa, we plot the data for $T > T_M = 2.4$ K, because the magnetic ordering appears below T_M. (b) The reciprocal susceptibility $1/\chi^{\mathrm{NMR}}$ as a function of temperature. χ^{NMR} is the susceptibility deduced from the K data. The solid and dotted lines represent the Curie-Weiss law with effective moments $\mu_{\mathrm{eff}} = 4.5\mu_B$ and $4.0\mu_B$, respectively. Inset: K vs χ plot with temperature as an implicit parameter for the paramagnetic state at ambient pressure. The solid line is a linear fit to the data. See text for details.

Now we discuss the results of the magnetic susceptibility in the pressure-induced paramagnetic state deduced from ^{115}In-NMR measurements. In general, the Knight shift is coupled to the uniform susceptibility χ by a linear relation. Figure 5(a) shows the temperature dependence of the Knight shift K measured at the In-site at different pressures. While K displays Curie-Weiss behavior in the paramagnetic state, its absolute value abruptly drops and shows temperature independent behavior, i.e., Pauli paramagnetic state below $T_V(P)$. At ambient pressure, the K vs χ plot for the paramagnetic state can be described by the relation $K(T) = -61.4\chi(T) + 0.2$ [see inset of Fig. 5(b)], where temperature is an implicit parameter. From the first term, we obtain the hyperfine coupling constant $H_{\mathrm{hf}} = -3.4$ kOe/μ_B, which is in good agreement with previous reports.[23,24] The second term is ascribed to the temperature independent components of χ and K, such as the van Vleck term. We note that K around 50 K is almost independent on pressure. This implies that H_{hf} is almost independent on pressure up to 2.6 GPa. We hence adopted the estimated value of H_{hf} at ambient pressure for the analysis of the K data at 2.0 and 2.6 GPa.

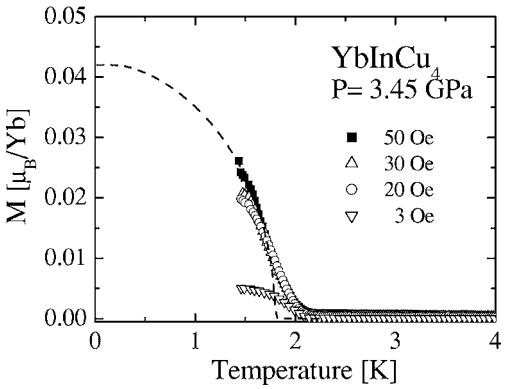

FIG. 6. Temperature dependence of the magnetization at 3.45 GPa. The data were taken according to process (iii) (see text) after magnetizing the sample at various fields of 3, 20, 30, and 50 Oe. The magnetization was calibrated by the method described in the text. The dotted line indicates the M-T curve calculated based on the simple mean-field approximation for $J = 7/2$.

As T_V is suppressed with increasing pressure, the temperature dependence of K solely follows the same Curie-Weiss line down to the lower $T_V(P)$ or T_M. The temperature dependence of the reciprocal susceptibility $1/\chi^{\mathrm{NMR}}$ is demonstrated in Fig. 5(b), where χ^{NMR} is the susceptibility deduced from the K data in the paramagnetic state at different pressures. It is known that χ in the paramagnetic state at ambient pressure exhibits a Curie-Weiss behavior with an effective moment near the full free-ion moment of Yb^{3+} ($4.54\mu_B$). The present result indicates that such well-localized character of the $4f$ electrons remains even in the pressure-stabilized paramagnetic state. This is consistent with the results of the spin-lattice relaxation time T_1 measurement on ^{63}Cu-site under pressure above P_C, i.e., from the temperature dependence of $1/T_1 \sim$ const. down to T_M, the T_1 relaxation process turns out to be dominated by the Curie-Weiss-type susceptibility.[13,14]

In the following, we discuss the magnitude of the ordered magnetic moment in the pressure region just above P_C. In order to calibrate the value of the magnetization in the present study, we used the jump in χ clearly observed at T_V. This height is known to be $\sim$0.04 emu/mol at ambient pressure from the conventional measurement using a SQUID magnetometer. By comparing this value with the magnetization jump at T_V and at ambient pressure measured in the DAC, the absolute values of the magnetization in the present experiment can be determined. Figure 6 shows the temperature dependence of the calibrated magnetization at 3.45 GPa, which was obtained after magnetizing the sample by different external fields of 3, 20, 30, and 50 Oe. Because the magnetization at low temperatures reveals the tendency toward saturating at 50 Oe, we tentatively deduce the magnitude of the ordered moment at the maximum field. To estimate the magnetization at 0 K by extrapolating the data for 50 Oe, we also plot in the figure a M-T curve which is calculated on the basis of simple mean-field approximation for $J = 7/2$. From this, the magnitude of saturated magnetic moment is roughly estimated to be $0.04-0.05\mu_B$. Inelastic neutron scattering experiments at ambient pressure indicate that, in the high tem-

perature phase of $YbInCu_4$, the crystalline electric field (CEF) ground state is a quartet Γ_8 and the first excited level is a doublet Γ_6 which is split by 3.2 meV.[25] The magnetic moment associated with the Γ_8 CEF state follows $\langle \mu_{\Gamma_8} \rangle = g_J \mu_B \langle \Gamma_8 | J_z | \Gamma_8 \rangle$ with $g_J = 8/7$. If we assume the same CEF regime and also well-localized $4f$ electrons in the high pressure ordered state of $YbInCu_4$, the expected full moment μ_{Γ_8} is $\sim 2.10 \mu_B$. Compared with this value, our present estimated value is significantly small.

A similar tiny ordered moment ($0.05 \mu_B$ at 1.2 GPa) was observed in 20% Y-substituted compound $Yb_{0.8}Y_{0.2}InCu_4$.[26] In this case, the possibility of itinerant ferromagnetism was discussed. For example, incomplete compensation of the $4f$ spin moments by the Kondo screening effect can resultantly cause small local moments. However it appears not to be the case for the high pressure phase of pure $YbInCu_4$, since no sign of Fermi liquid behavior associated with the Kondo effect was observed in the temperature dependences of K and $1/T_1$ in the pressure-stabilized phase, as mentioned above.

Very recently magnetic neutron scattering experiments were carried out in the pressure-induced ordered state of $YbInCu_4$.[27] According to this report, an ordered moment of about $0.6 \mu_B$ was observed above 2.5 GPa and below 2.5 K. This value is larger than our estimation by one order of magnitude. One of the possible explanations for this discrepancy is that the maximum field of 50 Oe applied to magnetize the sample possibly was not enough to form a single ferromagnetic domain structure. The previous ac-susceptibility measurement under pressure suggests that the field necessary for forming a complete single domain is possibly larger than 200 Oe.[10] Or the real magnetization at 0 K is perhaps larger than our extrapolation using a simple mean-field theory. Another peculiar behavior in the present magnetization measurement is that the magnetization at the lowest temperature has the largest value just above P_C and then monotonically decreases with increasing pressure, followed by saturating behavior above ~ 10 GPa, as shown in Fig. 2. This anomalous reduction in the magnetization with increasing pressure exceeds the experimental error, and is not associated with a structural phase transition as shown by the x-ray diffraction measurements. We, however, have no further information about its origin, namely whether it is ascribed to a decrease of ordered moment or to a change of spin structure. In order to clarify the mechanism, detailed analysis of the neutron scattering results is highly desired.

Finally, we discuss on the basis of these obtained results, possible electronic states of the $4f$ electrons in the pressure-stabilized phase of $YbInCu_4$. From the experimental fact that χ^{NMR} at 2.6 GPa follows the Curie-Weiss behavior with a large effective moment close to $4.5 \mu_B$ down to T_M, one expects that the pressure-induced paramagnetic state with well-localized $4f$ electrons directly transforms into the magnetically ordered phase as temperature decreases. If the application of pressure causes a change in the relative weight of the trivalent and divalent valence states of Yb ions in $YbInCu_4$, it should also lead to a change in the averaged ordered moment. Then, the interactions between local moments change, giving rise to a variation in T_M. The result demonstrated in Fig. 3 is contrary to this scheme, implying that application of pressure does not induce considerable changes in the hybridization between the $4f$ and conduction electrons. Many studies on $YbInCu_4$ indicate that the 4f electrons are in a well-localized state in the high temperature phase at ambient pressure. Therefore, it is reasonable to assume that the pressure-induced magnetic order mainly results from stabilizing this well-localized state down to the lowest temperature. We note that similar pressure behavior of the pressure-induced magnetic state has been observed in some Yb-based intermediate and heavy fermion compounds, e.g., $YbCu_2Si_2$ (Ref. 28) and $YbRh_2Si_2$ (Ref. 29): the sudden appearance of T_M above P_C and its weak pressure dependence. Such type of behavior therefore appears to be typical for pressure-induced magnetism in strongly correlated Yb-based compounds.

IV. CONCLUSIONS

We have investigated the pressure-induced magnetically ordered phase in $YbInCu_4$ by means of dc magnetization, NMR and x-ray diffraction measurements. The dc magnetization measurements give evidence for the appearance of spontaneous magnetization above 3 GPa, and a P-T phase diagram over a wide pressure range up to 27 GPa was established. In contrast to the drastic decrease of valence transition temperature T_V with increasing pressure, the magnetic ordering temperature T_M is found to be very weakly pressure dependent. We find no evidence for any structural phase transition up to 15 GPa within the experimental accuracy. The sudden appearance of T_M above critical pressure P_C and its weak pressure dependence are similar to those observed in some Yb-based strongly correlated electron systems. The analysis of the Knight shift obtained from ^{115}In-NMR measurements shows that there is no considerable pressure effect on the well-localized character of the $4f$ electrons in the paramagnetic state. We thus suggest that the pressure-induced magnetic order in $YbInCu_4$ results from stabilizing the well-localized electronic state of the $4f$ electrons down to the lowest temperature.

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