Spin freezing in the decagonal phase of the Al-Mn-Pd alloy system

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We report the results of measurements of the low-temperature specific heat $C_p(T)$ of annealed samples of decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$. A comparison with similar data, previously obtained for decagonal Al-Cu-Co quasicrystals, indicate a substantial excess specific heat $C_{ex}(T)$ in the Al-Mn-Pd material, most likely due to magnetic degrees of freedom. The temperature dependence of $C_{ex}(T)$ suggests a spin freezing phenomenon at $T_f = 12$ K, which is confirmed by the temperature dependence of the third-order magnetic susceptibility $\chi_3(T)$ and the time dependence of the remanent magnetization well below T_f.

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I. INTRODUCTION

Although in recent years considerable attention has been paid to experimental and theoretical studies of magnetic properties of thermodynamically stable quasicrystals, many questions concerning the nature of magnetism in these phases remain open.[1] For stable quasicrystals, different types of magnetic behavior have previously been observed, ranging from diamagnetism to a spin-glass-type freezing of magnetic moments.[2–8] Often the variation of the magnetic susceptibility versus temperature is indicative of a combination of a diamagnetic background and either a spin-glass-type or a Curie–Weiss type paramagnetic contribution at low temperatures (see, for example, Refs. 6, and 8–10).

Among the stable icosahedral phases, intrinsic spin-glass-type behavior has previously been reported for Al-Mn-Pd (Ref. 6) and *RE*-Mg-Zn (*RE* = Gd, Dy, Tb, Ho, Er).[7,8] The freezing of magnetic moments in the *RE* materials is reminiscent of that observed in the axial spin-glass systems $Y_{1-x}Tb_x$ and $Y_{1-x}Gd_x$.[11] For icosahedral *RE*-Mg-Zn, crystal-electric-field effects lead to a significant local magnetic anisotropy at the sites of the heavy rare-earth ions that have nonzero orbital angular momentum (Dy, Tb, Ho, and Er). The local anisotropy manifests itself, for example, by the higher freezing temperatures T_f of $(Y_{1-x}Tb_x)$-Mg-Zn solid solutions than those for the $(Y_{1-x}Gd_x)$-Mg-Zn alloys with the same heavy rare-earth concentrations x.[8]

Even more intriguing is the spin-glass behavior of the icosahedral Al-Mn-Pd quasicrystals. For icosahedral $Al_{70}Mn_9Pd_{21}$, the low-temperature saturation magnetization M_s, magnetic specific heat $C_m(T)$, and nuclear hyperfine specific heat $C_N(T)$, all indicate that a very low fraction of magnetic moments, of the order of 1% of all Mn atoms, is involved in the spin-glass freezing transition,[6] suggestive of the existence in this material of two classes of Mn sites—magnetic and nonmagnetic. We note here in passing that this is quite in contrast to canonical spin-glass systems $Cu_{1-x}Mn_x$ and $Au_{1-x}Mn_x$ where *all* Mn ions carry a magnetic moment. For icosahedral Al-Mn-Pd, both the fraction of magnetic Mn ions and the freezing temperature T_f increase rapidly with increasing Mn concentration. For example, T_f is enhanced by a factor of 7 when the Mn content is raised from 9 to 11 at %.[6,12] For thermodynamically stable icosahedral phases, a further enhancement of the Mn concentration is limited by the phase equilibria in the Al-Mn-Pd alloy system.[13,14] Related studies of the decagonal Al-Mn-Pd phase, which contains twice as much Mn as the corresponding stable icosahedral phase, are therefore of interest. While thermal and magnetic properties of icosahedral Al-Mn-Pd have previously been studied in detail experimentally,[6] analogous information for the corresponding decagonal phase is still scarce.[15]

Here we report the results of measurements of the specific heat $C_p(T)$ and the ac third-order magnetic susceptibility $\chi_3(T)$ of decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$, synthesized via coherent growth from a metastable icosahedral phase produced by melt spinning.[13,14] This technique allows to obtain single-phase samples of decagonal Al-Mn-Pd, which are free of linear phason strain. Our set of data, which also includes the result of a measurement of the time dependence of the remanent magnetization below the spin freezing temperature T_f, complements previous investigations of the magnetic susceptibility and magnetization of decagonal Al-Mn-Pd.[15]

II. SAMPLE, SYNTHESIS, AND STRUCTURAL CHARACTERIZATION

The decagonal phase in the Al-Mn-Pd alloy system can only be obtained via a solid-state transformation involving one or more of the following phases: the icosahedral Al-Mn-Pd phase, the orthorhombic H phase with approximate composition $Al_{71}Mn_{25}Pd_4$, and the cubic γ phase with approximate composition $Al_{60}Mn_{15}Pd_{25}$.[16] The decagonal phase has a very narrow existence region near $Al_{69.8}Mn_{18.1}Pd_{12.1}$.[13] Quite in contrast, the icosahedral phase, the H phase, and the γ phase all have extended regions of stability. Depending on the cooling rate, these phases can be obtained in a metastable state with varying chemical composition, leading to strongly segregated as-cast ingots. Therefore, the synthesis of the decagonal phase in an ingot with nominal composition $Al_{69.8}Mn_{18.1}Pd_{12.1}$ requires long-range diffusion of all three constituent elements. Annealing at approximately 800 °C for up to several weeks forms a decagonal phase of low quality exhibiting strong linear phason strain.[16–21] This is mainly because a segregated ingot cannot attain its equilibrium state. In principle, the equilibrium state can be reached by annealing at a temperature just below the melting point. The decagonal Al-Mn-Pd phase, however, de-

composes at approximately 865 °C into a nanocrystalline texture of a pseudodecagonal crystalline approximant phase with a solidus temperature of 896 °C,[13,14,17] which sets the upper limit of an annealing temperature to about 850 °C. Correspondingly, ingots annealed even at the highest possible temperature usually contain decagonal quasicrystal grains with domains that differ by the strength of linear phason strain.

Decagonal quasicrystals of high and rather uniform structural quality may be obtained by annealing tapes produced by rapidly quenching an $Al_{69.8}Mn_{18.1}Pd_{12.1}$ melt.[14,18] Melt-spun tapes quenched at a spinning velocity in the range between 20 and 30 m/s contain only a metastable icosahedral phase with the nominal chemical composition. Since $Al_{69.8}Mn_{18.1}Pd_{12.1}$ is the equilibrium composition of the decagonal phase, the icosahedral phase can be transformed into the decagonal phase without long-range diffusion. Annealing between 800 and 830 °C leads to a precipitation of the decagonal phase from the supersaturated icosahedral phase. The growth occurs in a coherent manner, i.e., the tenfold axis and a twofold axis of the decagonal phase are parallel to a fivefold axis and a twofold axis of the icosahedral phase, respectively.[22,23] Although the decagonal Al-Mn-Pd phase produced via coherent growth is free of linear phason strain, it is still perturbed by random phason strain. We note in passing that while linear phason strain breaks the decagonal symmetry, random phason strain retains on the average the decagonal symmetry.

Our sample of the decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$ phase was synthesized using a conventional melt-spinning technique with a wheel speed of 30 m/s. The as-quenched tapes were annealed at 820 °C for 4 days. Scanning electron microscopy and x-ray microanalysis of the annealed tapes revealed the presence of trace amounts of the orthorhombic H phase on the side of the tapes that was adjacent to the wheel while quenching the melt. Typically, the thickness of the H-phase spots is well below 1 μm, and we estimate the maximum content of the H phase in the tapes to be between 0 and 2 vol %. This estimate is in agreement with the results of the powder x-ray investigation, which did not reveal the presence of any phases other than the decagonal phase. From the powder x-ray-diffraction pattern, the lattice constant for the periodic direction (i.e., along the decagonal axis) was determined to be (1.2557 ± 0.0003) nm. A subsequent transmission electron microscopy investigation also indicated that the tapes contain practically 100% of the decagonal phase. A typical selected area electron diffraction (SAED) pattern obtained from the annealed tape with the decagonal axis oriented parallel to the electron beam is shown in Fig. 1. In this SAED pattern only the very weak diffraction spots are slightly shifted from the ideal positions that are expected for a decagonal phase practically free of random (as well as linear) phason strains. Only a weak diffuse background can be recognized, especially in the region close to the central beam. In summary, the average tenfold symmetry of the SAED pattern indicates that the decagonal phase is free of linear phason strains, and thus reflects a decagonal phase in the Al-Mn-Pd system of rather high structural perfection. For an additional characterization by high-resolution transmission electron microscopy images of our sample of decagonal Al-Mn-Pd quasicrystals, the reader is asked to consult Ref. 23.

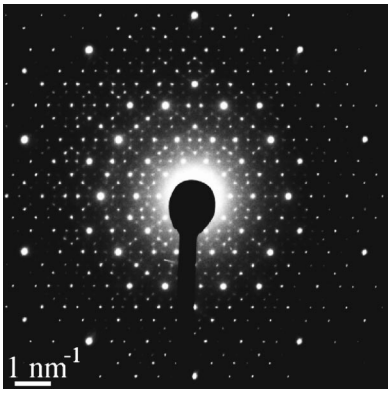

FIG. 1. An SAED pattern obtained from the decagonal phase in the annealed $Al_{69.8}Mn_{18.1}Pd_{12.1}$ tape. The tenfold axis was oriented parallel to the electron beam.

Bulky samples for measurements of physical properties were prepared by packing the necessary amounts of flakes into a steel container, and subsequently the material was pressed into a disk-shaped specimen. The resulting disks remained compact also after relieving the pressure.

III. EXPERIMENTS

The specific heat $C_p(T)$ was measured in the temperature range between 1.7 and 30 K using a conventional relaxation-type method. A sapphire disk onto which a heater and a Ge-Au thermometer had been evaporated was weakly thermally coupled to a heat sink held at constant temperature. The disk-shaped sample, prepared as mentioned above, was mounted on the sapphire disk using a thin layer of Apiezon grease.

The ac third-order magnetic susceptibility $\chi_3(T)$ was measured in the temperature range between 8 and 16 K, using an ac magnetometer probing χ_3 directly.[24] The primary coil of the magnetometer was driven at the reference frequency f by the internal oscillator of a phase-sensitive amplifier through a bipolar voltage-to-current converter. The amplitudes of the in-phase component V_3' and the out-of-phase component V_3'' of the third harmonic of the voltage induced in the secondary coil are proportional to the real part χ_3' and the imaginary part χ_3'' of the complex third-order susceptibility, respectively, and they were measured by the phase-sensitive detector, which was set to detect the response at $3f$. The time dependence of the remanent magnetization at low temperatures was monitored with a commercial SQUID magnetometer.

IV. RESULTS, ANALYSIS, AND DISCUSSION

A. Specific heat and magnetic properties

Our specific heat $C_p(T)$ data of decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$ is shown on a double-logarithmic plot in Fig. 2. Also the $C_p(T)$ data of nonmagnetic decagonal

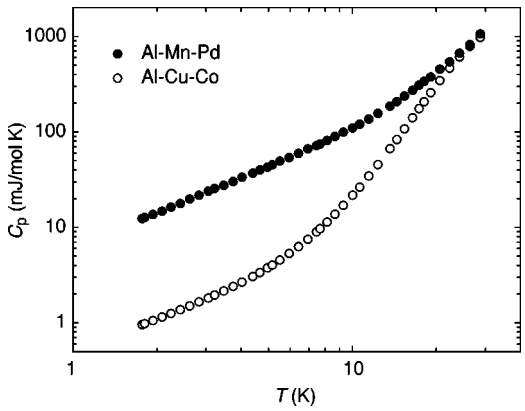

FIG. 2. Specific heat C_p of decagonal Al-Mn-Pd as a function of temperature T on logarithmic scales between 1.7 and 30 K. For comparison, we also show previously published $C_p(T)$ data of nonmagnetic decagonal Al-Cu-Co (Ref. 25).

$Al_{65}Cu_{20}Co_{15}$, previously reported by some of us in Ref. 25, are displayed in Fig. 2 as a reference. A comparison of these two datasets suggests that for decagonal Al-Mn-Pd, in the temperature range below 20 K, $C_p(T)$ is dominated by an excess specific-heat contribution $C_m(T)$ due to excitations of magnetic degrees of freedom, thus complicating a direct analysis of the specific-heat data of this quasicrystalline phase. For decagonal Al-Mn-Pd, the magnetic specific heat C_m may be obtained by subtracting estimates of the electronic term $C_{el}(T)$ and the lattice term $C_{ph}(T)$, established from the results of specific-heat measurements on the nonmagnetic decagonal Al-Cu-Co phase. In the temperature range between 1.7 and 10 K the specific heat of decagonal $Al_{65}Cu_{20}Co_{15}$ is an order of magnitude smaller than that of decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$. Therefore, below 10 K, $C_m(T)$ may be evaluated fairly accurately. Figure 3 shows the magnetic specific heat $C_m(T)$ of decagonal Al-Mn-Pd, as established in the way described above. In the inset of Fig. 3, we plot C_m as a function of T on logarithmic scales. Between 1.7 and 4 K, the temperature variation of C_m may very well be represented by a power law T^a with $a=1.22\pm0.01$. Our third-order magnetic-susceptibility data, presented and discussed below, reveals that decagonal Al-Mn-Pd undergoes a spin-glass-type freezing of magnetic moments at $T=12$ K (see Sec. IV B). In relation with the $C_m(T)$ data we recall that for $T \ll T_f$, a spin-glass magnetic contribution to the specific heat is expected to vary as T^a, where the exponent a usually takes values between 1.2 and 1.7.[26] The magnetic contribution C_m to the specific heat reaches a maximum at a temperature of 17 K, i.e., 40% higher than the freezing temperature T_f, which is typical of a spin glass.[27] In Fig. 4 we show the ratio $C_m(T)/T$ as a function of temperature T. We note that the maximum of $C_m(T)/T$ vs T is close to T_f, indicating that the freezing of the spins at T_f is associated with the maximum in the loss rate of magnetic entropy dS_m/dT. While this distinctive behavior of the magnetic specific heat C_m is not typical for spin-glass systems, close proximities of the C_m/T vs T maxima to T_f have previously been reported for metallic spin glasses $Cu_{1-x}Mn_x$, $Pd_{1-x}Mn_x$, icosahedral $Al_{70}Mn_9Pd_{21}$, and moderately disordered $Yb_5Pt_6In_{16}Bi_2$.[6,28–30]

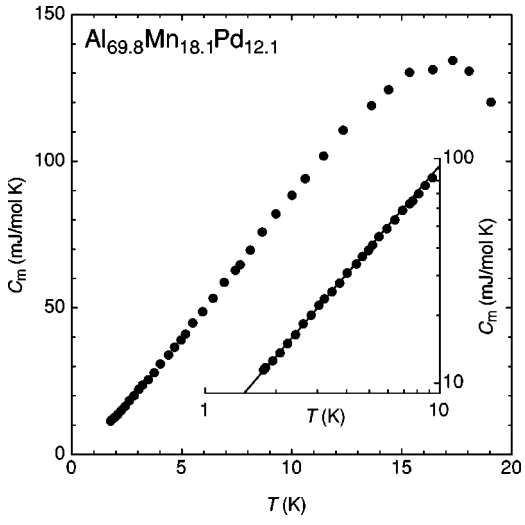

FIG. 3. Estimate of the magnetic contribution $C_m(T)$ to the specific heat of the decagonal Al-Mn-Pd phase. Inset: C_m versus T between 1.7 and 10 K plotted on logarithmic scales; the solid line is the power-law approximation to the data between 1.7 and 4 K (see text).

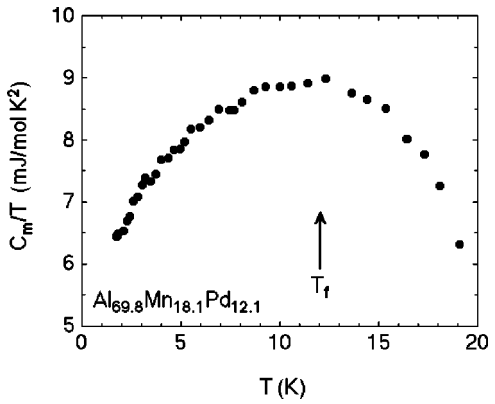

FIG. 4. C_m/T versus T of decagonal Al-Mn-Pd. The spin-glass-type freezing temperature T_f is indicated by an arrow.

The temperature dependence of the magnetic entropy $\Delta S_m(T)$, obtained from the magnetic contribution $C_m(T)$ to the specific heat via the equation

$$\Delta S_m(T) = \int_0^T \frac{C_m}{T'} dT', \tag{1}$$

is displayed in Fig. 5. For this calculation, C_m was extrapolated to zero temperature assuming a power-law variation $C_m(T) \propto T^{1.22}$ below 1.7 K (see Fig. 3). At the freezing temperature $T_f=12$ K, the magnetic entropy ΔS_m reaches 0.517 J/mol Mn K. For spin glasses, only between 22% and 30% of the magnetic entropy ΔS_m is normally developed below the freezing temperature,[31] reflecting a considerable short-range order of magnetic moments above T_f. We thus estimate the total magnetic entropy ΔS_m of decagonal Al-Mn-Pd to be between 1.72 and 2.35 J/mol Mn K. Assuming

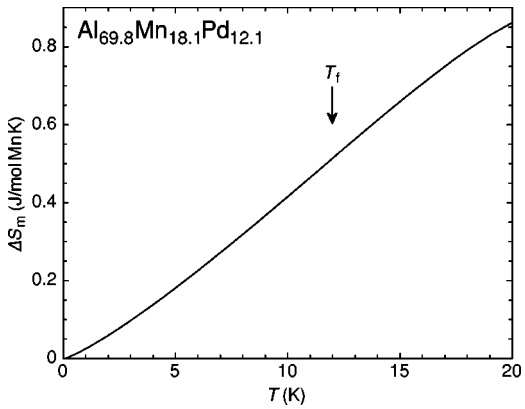

FIG. 5. The magnetic entropy ΔS_m of decagonal Al-Mn-Pd as a function of temperature T between 0 and 20 K.

that each Mn ion is trivalent, i.e., carries a spin $S=2$, leads to an expected loss of magnetic entropy of $\Delta S_m = 13.4$ J/mol Mn K. Our experimental ΔS_m values are distinctly smaller, thus suggesting that a large part of the Mn ions in decagonal Al-Mn-Pd carry no magnetic moment at low temperatures.

B. Magnetization

A useful probe that allows a rigorous identification of the freezing transition to the spin-glass state is the third-order magnetic susceptibility $\chi_3(T)$ that may be defined in terms of an applied magnetic field H and magnetization M as

$$M(T)=\chi(T)H+\chi_3(T)H^3+O(H^5). \tag{2}$$

For spin glasses, $\chi_3(T)$ is negative, and it diverges at the freezing temperature T_f.[32] We have measured the ac third-order magnetic susceptibility $\chi_3(T)$ of decagonal Al-Mn-Pd in the temperature range between 8 and 16 K. The real part $\chi_3'(T)$ of the complex third-order magnetic susceptibility shown in Fig. 6 displays a distinct negative anomaly, thus confirming the spin-glass nature of the freezing transition.

A characteristic feature of the irreversible behavior of the magnetization in a spin-glass system is the slow variation of

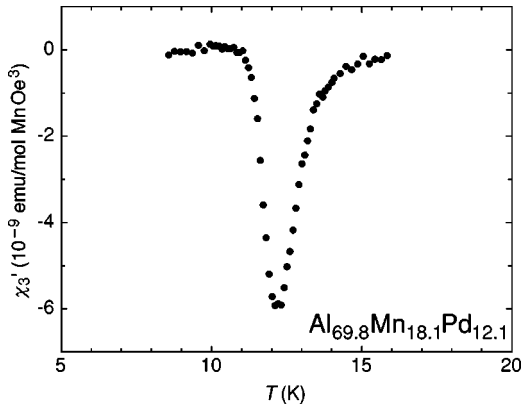

FIG. 6. The real part χ_3' of the complex ac third-order magnetic susceptibility of decagonal Al-Mn-Pd as a function of temperature T.

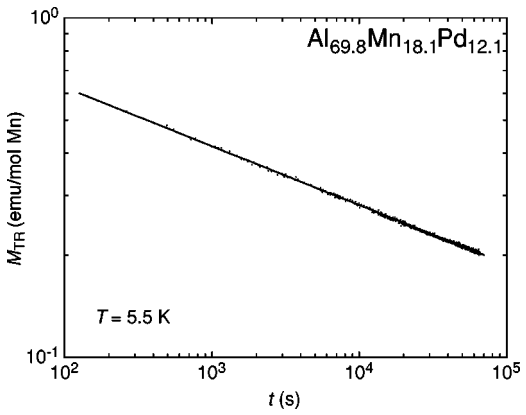

FIG. 7. The variation of the ''thermoremanent'' magnetization M_{TR} at $T=5.5$ K, plotted as a function of time t on logarithmic scales. The line is the fit to the $M_{TR}(t)$ data using Eq. (3) (see text).

a ''remanent'' magnetization with time.[27] We have measured the time dependence of the ''thermoremanent'' magnetization $M_{TR}(t)$ of decagonal Al-Mn-Pd over 2.5 decades in time after slowly cooling the sample from 50 to 5.5 K in an applied magnetic field of 55 kOe and subsequently removing the field. Our $M_{TR}(t)$ data shown in Fig. 7 can adequately be described by a power law

$$M_{TR}(t)=mt^{-a}, \tag{3}$$

implying that the relaxation involves a distribution of activation energies. Equation (3) has originally been proposed for spin glasses by Binder and Schröder (Ref. 33), based on the results of Monte Carlo simulations for square Ising lattices with random nearest-neighbor interactions. A fit of Eq. (3) to the $M_{TR}(t)$ data yields $m=1.39\pm0.01$ and $a=-0.174\pm0.001$. The value of the exponent a is consistent with analogous values previously reported for various spin-glass systems.[27] The slow relaxation of $M_{TR}(t)$ is a general observation for spin glasses, which reflects the nature of the low-temperature state of these systems, with multiple configurations separated by energy barriers of varying heights. The variation of the remanent magnetization with time is not universal, however, and it appears to vary from one spin-glass system to another.[34]

V. SUMMARY

The low-temperature specific heat and the ac third-order magnetic susceptibility indicate a spin freezing phenomenon in annealed single-phase material of decagonal $Al_{69.8}Mn_{18.1}Pd_{12.1}$ at $T_f=12$ K.

Results of measurements of the time dependence of the ''thermoremanent'' magnetization M_{TR} and also the slow variation of $M_{TR}(t)$, all measured below T_f, are typical of canonical spin glasses, thus giving additional support to our classification of the low-temperature state of decagonal Al-Mn-Pd.

The entropy variation that is related with the spin freezing transition indicates that a substantial fraction of the Mn ions carries no magnetic moment at low temperatures. Satoh *et al.*[15] reported that the fraction of magnetic Mn ions in

decagonal Al-Mn-Pd was 2.5 at % Mn, i.e., 14% of all Mn atoms. By contrast, recent nuclear-magnetic-resonance measurements indicate that as much as 9 at % Mn carries a magnetic moment, i.e., 50% of all Mn atoms.[35]

The ratio T_f/x, a quantity that reflects the strength of interaction between magnetic moments, lies in the range between 130 and 480 K. The most likely value for decagonal Al-Mn-Pd is therefore lower but nevertheless quite close to the corresponding value of icosahedral $Al_{70}Mn_9Pd_{21}$ and clearly above that observed for the quasicrystalline *RE*-Mg-Zn (*RE*=Dy, Tb, Ho, Er) alloys. In icosahedral $Al_{70}Mn_9Pd_{21}$ a value of $T_f/x=450$ K may be inferred from the results of our measurements of thermal and magnetic properties (see Ref. 6). By comparison, quasicrystalline *RE*-Mg-Zn (*RE*=Dy, Tb, Ho, Er) alloys are characterized by distinctly weaker interaction strengths, reflected by the T_f/x values ranging from 15 to 70 K.[8] Values of T_f/x, which are comparable in magnitude with our findings for icosahedral and decagonal Al-Mn-Pd, have previously been reported for metallic spin-glass systems, such as $Cu_{1-x}Mn_x$, $Ag_{1-x}Mn_x$, and $Au_{1-x}Mn_x$, all with strong Ruderman-Kittel-Kasuya-Yosida exchange interactions among Mn moments (see Ref. 36).

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