



Perturbed angular correlation studies of ^{181}Ta hyperfine interactions in Hf–Ni and Zr–Ni compounds

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ABSTRACT

The hyperfine interaction experienced by ^{181}Ta nuclei in the intermetallic compounds ZrNi₅, HfNi₅, and Hf₂Ni₇ has been investigated by perturbed angular correlation (PAC) spectroscopy. At temperatures $T \geq 15$ K the ^{181}Ta angular correlation of appropriately annealed ZrNi₅ and HfNi₅ is unperturbed, indicating the absence of a magnetic hyperfine interaction. This observation rules out the possibility of spontaneous magnetic order of ZrNi₅ and HfNi₅ recently proposed in the literature. The temperature dependence of the electric quadrupole interaction of ^{181}Ta in Hf₂Ni₇ suggests the existence of a reversible phase transformation at $T \geq 500$ K.

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1. Introduction

Earlier studies of the magnetic properties of the Zr–Ni intermetallic compound ZrNi₅ have led to diverging conclusions: On one hand, magnetization measurements by Amamou et al. [1] and electronic structure calculations by Turek et al. [2] have provided no evidence for ferromagnetic order in ZrNi₅ at temperatures $T \geq 4.2$ K. Very weak itinerant ferromagnetism has been found [3] in metastable Zr–Ni and Hf–Ni alloys only beyond a critical Ni concentration of about 90 at.% (ZrNi₁₀). On the other hand, Drulis et al. [4] have concluded from magnetization vs. temperature data that ZrNi₅ is a rather strong ferromagnet with a Curie temperature of 647 K.

Nuclei in ferromagnetic solids experience a magnetic hyperfine field B_{hf} (Ref. [5]). Ferromagnetic order can therefore be detected by observing the resulting Larmor precession of nuclear magnetic moments with frequency $\omega_m = 2\pi\nu_m = g\mu_N B_{\text{hf}}/\hbar$ (g denotes the nuclear g factor). In this communication we report a search for spontaneous magnetic order of ZrNi₅ and HfNi₅ by looking for the existence of a magnetic hyperfine field at the Zr(Hf) site.

The search was carried out with the perturbed angular correlation (PAC) technique [6]. The angular correlation of two successive γ -rays of a $\gamma\gamma$ -cascade in nuclear decay may be modulated in time by hyperfine interactions in the intermediate state of the cascade. The observation of the time-dependence of

an angular correlation therefore provides information on magnetic and electric hyperfine interactions in condensed matter.

In the present study, the isotope ^{181}Ta was used as nuclear probe. Apart from the favourable nuclear parameters of its $\gamma\gamma$ -cascade (anisotropy, half life, and nuclear moments), the fact that the excited states of ^{181}Ta are populated by the β^- -decay of ^{181}Hf ($T_{1/2} = 42$ d) makes this isotope the ideal probe for PAC studies of Hf and Zr compounds. The recoil involved in the β^- -decay of ^{181}Hf is too small (< 6 eV; Ref. [7]) to dislocate the decaying nucleus from its lattice position, and one can therefore be sure that in Hf compounds the PAC probe ^{181}Ta resides on regular Hf sites. The same argument holds for ^{181}Hf -doped Zr compounds where – due to the pronounced chemical similarity of Hf and Zr – ^{181}Hf substitutes Zr atoms. These considerations have motivated ^{181}Ta PAC studies of numerous Hf(Zr) compounds, among them several Hf(Zr)–Ni intermetallics [8–13].

The Hf(Zr) site (4a) of the AuBe₅-type structure of Hf(Zr)Ni₅ (space group $F-43m$) has cubic symmetry point with zero electric field gradient (EFG). A nuclear quadrupole interaction for ^{181}Ta on the Hf(Zr) site can thus be excluded. In case the host compound Hf(Zr)Ni₅ presents spontaneous magnetic order, one therefore expects a perturbation by a pure magnetic hyperfine interaction, in the absence of magnetic order the angular correlation will be unperturbed, i.e. constant in time.

At the Ni-rich end the phase diagrams of the binary Zr–Ni and Hf–Ni systems [14] are rather similar. Both show the phases Hf(Zr)Ni₅ and Hf(Zr)₂Ni₇. The latter melts congruently at 1440(1480)°C and Hf(Zr)Ni₅ forms through the peritectic reaction $L + \text{Hf}(\text{Zr})_2\text{Ni}_7 \leftrightarrow \text{Zr}(\text{Hf})\text{Ni}_5$ 1300(1240)°C. An eutectic reaction $L \leftrightarrow \gamma + \text{Hf}(\text{Zr})\text{Ni}_5$ with γ the terminal solution of Hf(Zr) in Ni occurs at 1170(1190)°C. A sample of Hf(Zr)Ni₅ produced in

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non-equilibrium conditions, e.g., by quenching from the melt, may therefore contain the phases $\text{Hf}(\text{Zr})\text{Ni}_5$ and $\text{Hf}(\text{Zr})_2\text{Ni}_7$ and precipitates of fcc Ni. For the identification of eventual $\text{Hf}(\text{Zr})_2\text{Ni}_7$ contributions to the PAC spectra of $^{181}\text{Ta}:\text{Hf}(\text{Zr})\text{Ni}_5$, we have also studied the hyperfine interaction of ^{181}Ta in Hf_2Ni_7 as a function of temperature. According to Eshelman and Smith [15], Zr_2Ni_7 crystallizes in a monoclinic crystal structure with $C2/m$ space group symmetry. Dattagupta and Schubert [16] have shown Hf_2Ni_7 to be isotopic to Zr_2Ni_7 .

2. Experimental

2.1. Sample preparation and characterization by X-ray diffraction

PAC samples of ZrNi_5 , HfNi_5 , and Hf_2Ni_7 were produced by melting stoichiometric amounts of the metallic components –

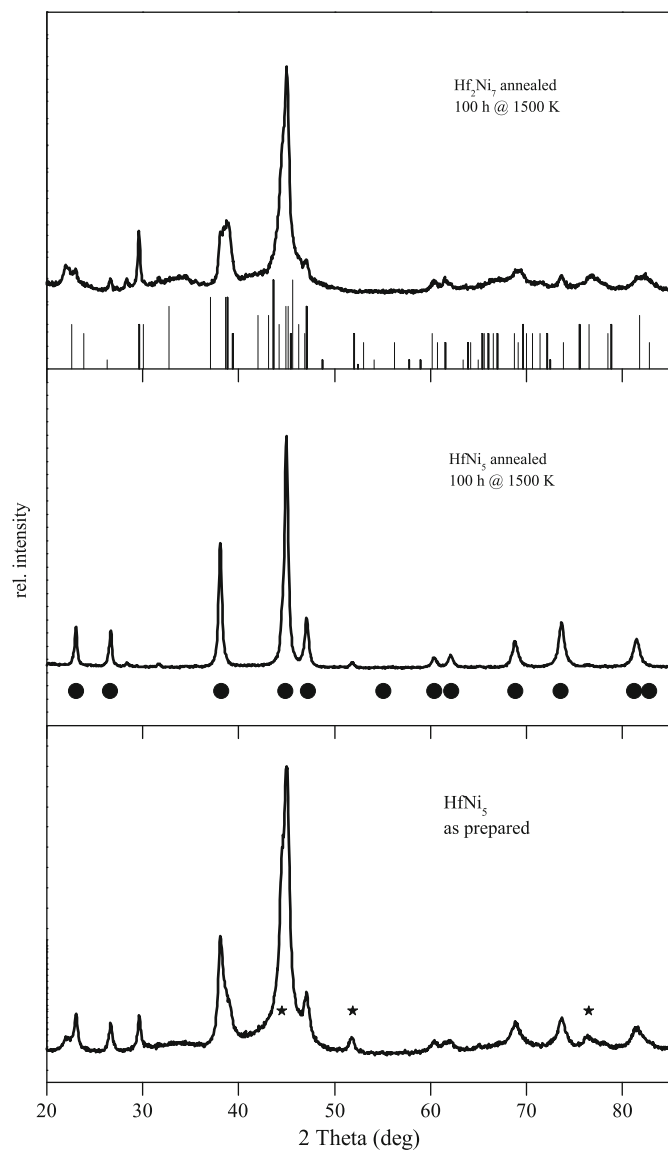


Fig. 1. X-ray diffraction pattern of HfNi_5 in the as-prepared state and after annealing for 100 h at 1500 K, taken at 300 K with Cu K_α radiation. The full points in the middle section mark the main reflections of ZrNi_5 observed by Smith and Guard [18]. The full stars in the bottom section correspond to reflections of fcc Ni. The top-most section shows the diffraction pattern of annealed Hf_2Ni_7 with the vertical bars representing the diffraction diagram of Hf_2Ni_7 reported by Dattagupta and Schubert [16].

together with about 0.1 at.% of radioactive ^{181}Hf metal – in an arc furnace under argon atmosphere. Inactive samples for X-ray diffraction studies were prepared in the same way. In the as-prepared state at room temperature the samples of ZrNi_5 and HfNi_5 were found to be ferromagnetic. The spontaneous magnetization disappeared after annealing for 100 h at 1500 K. The same observations have been reported by Kissell et al. [17]. No spontaneous magnetization was found for Hf_2Ni_7 .

Because its formation through a peritectic reaction, homogenisation of a sample of $\text{Hf}(\text{Zr})\text{Ni}_5$ rapidly cooled from the melt requires annealing at high temperatures. We have studied the effect of a high-temperature treatment of rapidly cooled $\text{Hf}(\text{Zr})\text{Ni}_5$ both with X-ray diffraction and perturbed angular correlations.

Fig. 1 shows the X-ray diffraction pattern of HfNi_5 in the as-prepared state and after annealing at 1500 K for 100 h. The pattern of annealed ZrNi_5 was identical to that of HfNi_5 . The spectra—taken at room temperature with K_α radiation—mainly consist of the pattern of a AuBe_5 -type compound and agree in the main features with the ZrNi_5 spectra reported by Smith and Guard [18], Gachon et al. [19] and Drulis et al. [4]. The lattice parameter derived from the spectra in Fig. 1 ($a=0.6686(10)$ nm and $0.6706(10)$ nm for HfNi_5 and ZrNi_5 , respectively) also agree with the values previously reported [4,18,19] for ZrNi_5 .

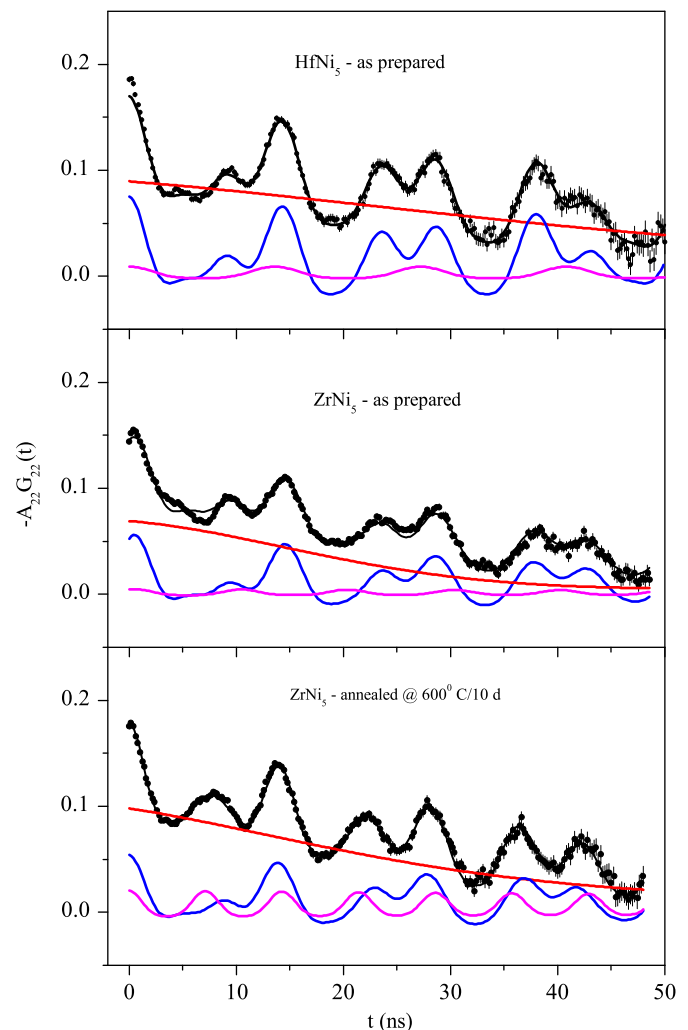


Fig. 2. PAC spectra of ^{181}Ta in HfNi_5 and ZrNi_5 at 295 K in the as-prepared state. The samples were produced by arc-melting and rapid cooling from the melt. The bottom-most spectrum was obtained after annealing ZrNi_5 for 10 d at 900 K.

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