Interplay between magnetism and superconductivity in HoNi$_2$B$_2$C revisited

E Alleno$^{1,4}$, S Singh$^{2,5}$, S K Dhar$^2$ and G André$^3$

1 ICMPE-CMTR, UMR 7182 CNRS—Université Paris 12, 2–8 rue H Dunant, 94320 Thiais, France
2 TIFR, Homi Bhabha Road, Mumbai 400005, India
3 LLB, CEA-CNRS Saclay, 91191 Gif Sur Yvette Cedex, France
E-mail: eric.alleno@icmpe.cnrs.fr

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Abstract. In this work the magnetic and superconducting properties of HoNi$_2$B$_2$C were investigated by using powder neutron diffraction and the specific heat and upper critical field ($H_{c2}$) measurements as a function of temperature. Below $T = 8$ K, three distinct anomalies at the temperatures $T_N = 5.2$ K, $T_{H1} = 5.6$ K and $T_M = 6.0$ K were observed in the specific heat of HoNi$_2$B$_2$C, as reported in the literature. Our neutron data confirm the transitions to the Néel structure ($q_N = c^*$) at $T_N$ and to the modulated structure ($q_M = 0.586a^*$) at $T_M$. The peak at $T_{H1} = 5.7$ K in the specific heat data, whose exact nature was not known hitherto, is now attributed to the onset of a $q_{H1} = 0.905c^*$ magnetic helical structure as seen in our neutron data. Comparison between the thermal evolution of the magnetic structures and the temperature dependence of the upper critical field confirms that the first $H_{c2}(T)$ depression at 6.1 K arises from the $q_M = 0.586a^*$ modulated magnetic structure. The second depression in $H_{c2}(T)$ below 5.7 K can be ascribed to the $q_{H1} = 0.905c^*$ magnetic helical structure.

$^4$ Author to whom any correspondence should be addressed.
$^5$ Current address: Indian Institute of Science Education and Research, 900, NCL Innovation Park, Dr Homi Bhabha Road, Pune 411008, Maharashtra, India.
1. Introduction

The coexistence of superconductivity and magnetic order in the rare-earth (R)-nickel-borocarbides RNi$_2$B$_2$C (R = Dy, Ho, Er or Tm) is a rare phenomenon since these two long-range electronic states are mutually exclusive in most cases. In these compounds, the localized rare-earth 4f magnetic moments are coupled to the conduction electrons (mainly Ni 3d) by the direct s–f exchange interaction. The itinerant nature of the conduction electrons results in a magnetic coupling of the rare-earth magnetic moments mediated by the indirect Rudermann–Kittel–Kasuya–Yosida interaction. In these compounds this leads to magnetic transition temperatures ($T_N$) comparable to superconducting transition temperatures ($T_c$) (a few kelvins) and to an interplay between magnetism and superconductivity [1].

This interplay is still a matter for discussion in the case of HoNi$_2$B$_2$C ($T_c$ = 8.5 K) because the latter displays the most complex magnetic phase diagram of the series. Three antiferromagnetic (AF) structures were detected in this tetragonal compound ($I 4/mmm$) by neutron diffraction in zero magnetic field [2–4]. Below $T_N = 5.3$ K, the Ho moments (m) that are aligned parallel in the (a, b) plane alternate antiferromagnetically along the c-direction with a commensurate propagation vector $q_N = e^c$. Above 5.3 K, this commensurate structure transforms into two incommensurate AF configurations that coexist: an amplitude modulated structure with $q_M = 0.585a^*$ and $m//b$ that disappears at $T_M = 6.0$ K and a helical structure with $q_{H2} = 0.915c^*$ and $m//(a, b)$ that persists up to approximately 8 K. Adding to this complexity, magnetic x-ray scattering combined with neutron scattering [5] revealed, in a very narrow temperature range (5.2–5.5 K), the existence of another helical structure with $q_{H1} = 0.905c^*$. In HoNi$_2$B$_2$C, the interaction of magnetism with superconductivity manifests itself as a strong depression of the upper critical field ($H_{c2}(T)$) as a function of temperature between 5 and 6 K [6–11], leading to a re-entrant or nearly re-entrant behavior in the thermal variations of resistivity and ac-susceptibility. Based on the fact that the $H_{c2}(T)$ local maximum occurs at $T_M = 6.0$ K, the depression in $H_{c2}(T)$ was attributed to the $q_M = 0.585a^*$ modulated structure [12–15]. Since $q_M$ is a nesting vector of the Fermi surface, the depression in $H_{c2}(T)$ caused by the modulated structure was suggested to arise from the Machida–Ramakrishnan

6 In [4], alternate spin arrangements were proposed for the $q_{H2}$ structure but the helical structure was finally the preferred one.

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mechanism [16, 17]. This scenario is plausible but it leaves aside the helical structure which was shown in a theoretical work by Amici–Thalmeier–Fulde [18] to also depress the superconducting order parameter. More recently, another calculation [19] of \( H_{c2}(T) \) in HoNi\(_2\)B\(_2\)C surprisingly ignored the incommensurate structures and ascribed the depression at 6.0 K to the \( q_N = c^* \) commensurate structure only. We report, in this paper, the specific heat \( C_p(T) \), elastic neutron scattering and \( H_{c2}(T) \) data as a function of temperature in the case of HoNi\(_2\)B\(_2\)C. Combining these sets of results, we found that the onset of the \( q_{HI} = 0.905c^* \) magnetic helical structure correlates with a reduction of \( H_{c2}(T) \) at 5.7 K.

2. Experiment

Four HoNi\(_2\)B\(_2\)C\(_{1.05}\) polycrystalline samples (2.5 g each) were arc melted and annealed at 1100 °C for six days. Five per cent excess carbon was added to guarantee stoichiometric samples (for details of the preparation method, see [20]) and the samples will therefore be labeled HoNi\(_2\)B\(_2\)C. \(^{11}\)B isotope (Eurisotop, 97.5% isotopically enriched) was used to reduce the neutron absorption caused by \(^{10}\)B in natural boron. The lattice parameters of these four samples, as determined by x-ray powder diffraction, spanned the intervals 3.517 Å \( \leq a \leq 3.518 \) Å and 10.527 Å \( \leq c \leq 10.528 \) Å. The typical content of secondary phases for these samples was 1% Ni\(_2\)B and 3% HoB\(_2\)C\(_2\). Magnetization measurements (\( H = 10 \) Oe) in a superconducting quantum interference device (SQUID) confirmed that these four HoNi\(_2\)B\(_2\)C samples are superconducting at 8.2 K. Specific heat measurements (\( H = 0 \)) were carried out in a semiadiabatic, heat-pulse-type home-made calorimeter on \( \sim 1.5 \) g of sample mass, cut from one of the four buttons. As a preliminary, the specific heat of LaNi\(_2\)B\(_2\)C was measured with this setup and was subtracted to obtain the magnetic contribution in HoNi\(_2\)B\(_2\)C. We determined \( H_{c2}(T) \) in HoNi\(_2\)B\(_2\)C by carefully measuring several resistance versus field isotherms (\( R(H) \)) (not corrected from the demagnetization effects). These isotherms were measured in a PPMS (Quantum Design) in applied ac-current mode (four probes, \( J = 0.1 \) A cm\(^{-2}\), frequency = 107 Hz, fieldstep = 33 Oe) on a platelet-like specimen cut from the same button as the one used for the \( C_p(T) \) measurement. After completion of these measurements, the four samples were ground to a fine powder and mixed together to make a \( \sim 9 \) g sample, large enough to ensure a good neutron scattering signal. This neutron experiment was performed at the Laboratoire Léon Brillouin (CEA-CNRS Saclay, Gif-sur-Yvette, France) using the G4.1 2-axis multidetector diffractometer (\( \lambda = 2.427 \) Å) in the [1.4–10 K] temperature range. The 9 g HoNi\(_2\)B\(_2\)C sample was held in a vanadium can (radius = 4 mm) and placed in an He cryostat.

3. Results and discussion

3.1. Neutron elastic scattering

A portion of the neutron powder pattern of HoNi\(_2\)B\(_2\)C measured at 5.7 K is plotted in figure 1. At this temperature, the scattered intensity associated with the transverse modulated and the helical structures as well as the pre-transitional scattering associated with the Néel structure can easily be observed [2–4]. The magnetic peaks can be indexed by taking \( q_N = c^* \) for the Néel structure, \( q_{H2} = 0.911c^* \) for the helical structure and \( q_M = 0.586a^* \) for the modulated structure. Faint third-order peaks of the helical structure (\( 3q_{H2} \)) can also be seen between \( \sim 6 \) and 5 K and this
suggests a possible bunching of this structure. The second helical structure previously revealed by magnetic x-ray and neutron scattering [6] cannot be seen in figure 1 at 5.7 K. Hence, we initially considered only one helical structure for every temperature and fitted the helical peaks with a single Gaussian. However, when plotting (not shown) the corresponding single $q$-vector and width as a function of temperature, an anomaly—a strong increase—could be detected at 5.7–5.6 K in both quantities. These anomalies were confirmed by a careful examination of the helical peaks as displayed in figure 2. One can easily notice that a shoulder develops on the inner side of each of the most intense helical peaks ($q_{H2}$ and (002)-$q_{H2}$) below 5.7 K. This corresponds to the appearance of the $q_{H1}$ = 0.905$c^*$ structure and to the shift to higher $q$-vector values of the $q_{H2}$ structure as revealed by magnetic x-ray scattering [5]. It is possible to fit two Gaussians to our data, but due to their poor resolution, it is mandatory to make assumptions on their width and position to obtain consistent results across the set of temperatures. We chose to keep their width constant to the value found above 5.7 K (0.027 r.l.u.). We fixed their positions to the values found in [5] as temperature varies: $q_{H1}$ and $q_{H2}$, respectively, increase from 0.903$c^*$ to 0.905$c^*$ and from 0.911$c^*$ to 0.923$c^*$ when $T$ decreases from 5.7 to 5.1 K. The only free fitted parameters were the intensities of the two Gaussians, which could hence be extracted. An example of such a fit, performed at 5.3 K on the $q_{H}$ manifold, is presented in the inset of figure 2. Above 5.6 K, attempts to fit the data with two Gaussians resulted in negative intensities for the $q_{H1}$ structure and only one Gaussian corresponding to the $q_{H2}$ structure could be fitted to our data. Below 5.6 K, positive intensities were obtained for both structures. This is the first time this extra helical structure is reported in a neutron powder work. We could also detect a faint third-order peak for the second helical structure ($3q_{H1}$) at 5.3 and 5.4 K and this suggests that this structure is also bunched. The integrated intensities ($I$) of the four magnetic structures are plotted as a function of temperature in figure 3. For the $q_{N}$ and $q_{M}$ magnetic structures, their intensity was derived.

Figure 1. Portion of the neutron powder pattern of HoNi$_2$B$_2$C recorded at 5.7 K. $q_{N} = c^*$, $q_{H2} = 0.911c^*$ and $q_{M} = 0.586a^*$ are the propagation vectors of the Néel, the helical and the transverse modulated structures, respectively.
Figure 2. Enlarged view of the two most intense helical peaks in HoNi$_2$B$_2$C as a function of the diffusion vector in r.l.u. for several temperatures. A second helical structure grows at $q_{H1} = 0.903c^*$ below 5.6 K, while the other $q_{H2} = 0.911c^*$ helical structure shifts to larger $q$-values upon cooling. The bottom arrows point to the $q_{H1}$ position, while the two top arrows represent the range of $q$-values of the $q_{H2}$ structure. The central peak corresponds to pre-transitional scattering associated with the Néel structure. The inset shows a fit of the data at 5.3 K by two Gaussians, with $q_{H1} = 0.904c^*$ and $q_{H2} = 0.921c^*$.

from a simple integration of their most intense peak. At temperatures below 5 K, only the Néel structure exists and its intensity strongly decreases above this temperature. A power law fit yields $T_N = 5.2 \pm 0.1$ K. Above $T_N$, this commensurate structure transforms into the $q_{H1} = 0.905c^*$ helical structure, the $q_{M} = 0.586a^*$ modulated structure and the other $q_{H2} = 0.923c^*$ helical structure. These three incommensurate magnetic structures nearly peak at the same temperature, e.g. 5.4 K, but their transition temperature is different. The $q_{H1}$ helical structure and the $q_{M}$ modulated structure both abruptly disappear at their transition temperatures, which are $T_{H1} = 5.6$ K and $T_{M} = 6.0$ K, respectively. The intensity of the other $q_{H2}$ helical structure slowly decreases and persists up to 8 K and no transition temperature can easily be defined. These results (temperature dependence and transition temperatures) are in very good agreement with the literature [2–4]. There is only a small difference between the transition temperatures $T_{H1}$ that we found and that reported in the magnetic x-ray scattering and neutron scattering experiments of Hill et al [5]: we found $T_{H1} = 5.6$ K, while $T_{H1} = 5.5$ K was reported in [5]. The small temperature difference (0.1 K) most likely arises from heating of the sample by the x-ray beam since the onset of the $q_{M}$ modulated structure is seen at $T_{M} = 5.9$ K in [5], in contrast to the $T_{M} = 6.0$ K derived from our neutron data and others' [3]. Table 1 summarizes all these transition temperatures.
Figure 3. Integrated intensities versus temperature of the most intense neutron magnetic peaks in HoNi$_2$B$_2$C. (A) The $q_M = 0.586a^*$ modulated structure. (B) The $q_{H1} = 0.903-0.905c^*$ helical structure. (C) The $q_{H2} = 0.911-0.923c^*$ helical structure. (D) The $q_N = c^*$ Néel structure.

3.2. Specific heat

The magnetic contribution to the specific heat for HoNi$_2$B$_2$C is plotted in figure 4 as a function of temperature. A large and sharp peak at $T_N = 5.2$ K and a relatively smaller peak at $T_{H1} = 5.7$ K, besides a small shoulder at $T_M = 6.0$ K, can easily be seen. The $T_N = 5.2$ K peak has been ascribed to the Néel transition [7, 8, 21]. Based on the anomalous in-plane anisotropy deduced from the specific heat data measured in applied magnetic fields at various orientations, Park et al [22] ascribed the weak $T_M = 6$ K shoulder to the onset of the modulated $a^*$ magnetic structure. The second peak at $T_{H1} = 5.7$ K (in figure 1) was also reported at the same temperature by Lin et al [8] but at a slightly lower temperature of 5.5 K by other groups [7, 21, 22]. Small differences in the carbon content or in the residual strains or defects (chemical or structural) from sample to sample are possible explanations for this small difference. Based
Table 1. Temperature of the characteristic features seen in the neutron scattered intensities, $C_p(T)$ and $H_{c2}(T)$ data. See the text for definitions.

| Temperature | Neutron $C_p$ | Temperature $H_{c2}$ |
|-------------|---------------|-----------------------|
| $T_N$ (K)   | 5.2           | $T_{min}$ (K) 5.3     |
| $T_{H1}$ (K)| 5.6           | $T_{bp}$ (K) 5.7     |
| $T_M$ (K)   | 6.0           | $T_{max}$ (K) 6.1     |

Figure 4. Thermal variation of the magnetic contribution to the specific heat in HoNi$_2$B$_2$C. The peaks labeled $T_N$ and $T_M$ correspond to the transition to the Néel state and the transition to the modulated structure, respectively. We ascribe the peak at $T_{H1}$ to the transition to the $q_{H1} = 0.905c^*$ helical state (see the text).

on its relative position to the other two anomalies at $T_N$ and $T_M$, this peak (labeled $T^*$ or $T_2$ in previous works) was believed to indicate a change in the $c^*$ helical state, [8, 21] and based on its magnetic field dependence and small in-plane anisotropy, it was ascribed to a transition from the $q_M$ modulated structure to the $q_{H2}$ helical state [22]. However, this last scenario is not compatible with the neutron data presented in figure 3, which show that, upon cooling, both these incommensurate magnetic states coexist and vanish at the same temperature $T_N = 5.2$ K. We rather suggest that it corresponds to the transition to the $q_{H1} = 0.905c^*$ helical structure as seen in our neutron data. As already discussed for the neutron part, the other magnetic helical structure with $q_{H2} = 0.923c^*$ does not display an onset but rather a progressive increase of the neutron scattered intensity upon cooling below 8 K and we believe that this leads to the absence of a corresponding peak in the specific heat data. These transition temperatures determined by specific heat are also shown in table 1 for comparison with the neutron values. There is a very good match between the two sets of data and this further supports our interpretation of the transition temperatures seen in specific heat.
3.3. \( H_{c2}(T) \) measurements

\( T_c = 8.3 \) K was obtained from a preliminary measurement of resistivity versus temperature for the HoNi\(_2\)B\(_2\)C sample. It is very close to the \( T_c = 8.2 \) K determined by SQUID magnetometry. In figure 5, a few examples of \( R(H) \) isotherms are displayed. The mid-point resistance field (\( H_{mid} \)) is determined from these \( R(H) \) data and we use \( H_{c2} = H_{mid} \) to define the upper critical field in agreement with previous works [9, 10]. The upper critical field \( H_{c2} \) for HoNi\(_2\)B\(_2\)C is plotted as a function of temperature in figure 6. \( H_{c2}(2 \) K\) = 5.2 kOe (not shown), in agreement with previous works on polycrystals [6, 23]. Usually, \( H_{c2}(T) \) for HoNi\(_2\)B\(_2\)C is reported to exhibit a maximum close to \( T_{max} = 6 \) K and a minimum close to \( T_{min} = 5 \) K. Our detailed measurements (figure 6) show that the situation is more complex. Upon cooling, \( H_{c2}(T) \) for HoNi\(_2\)B\(_2\)C weakly decreases between \( T_{max} = 6.1 \) and 5.7 K and displays a steep decrease only between \( T_{min} = 5.7 \) and 5.3 K. \( H_{c2}(T) \) for HoNi\(_2\)B\(_2\)C thus displays a ‘two-slope’ behavior, where \( T_{bp} = 5.7 \) K is a break-point temperature separating two regions differentiated by the extent of the superconductivity depression. These two slopes can also be observed in the \( H_{c2}(T) \) data of single crystals measured with \( \text{H/}[001] \) [9, 10], but their implication was not discussed. \( T_{min} \), \( T_{bp} \) and \( T_{max} \) are also shown in table 1.

3.4. Discussion

Magnetic phase diagram measurements [10, 11, 22] have shown that \( T_N \), \( T_{H1} \) (denoted \( T_2 \) in these previous works) and \( T_M \) vary by less than 0.1 K with the magnetic field when \( H < 2 \) kOe, thus permitting a direct comparison of the zero-field neutron \( I(T) \), \( C_p(T) \) and \( H_{c2}(T) \) data. The \( T_{max} \) and \( T_M \) coincidence has already been reported and commented on.
Figure 6. The upper critical field $H_{c2}(T)$ in HoNi$_2$B$_2$C. The temperatures of the maximum ($T_{\text{max}}$), the minimum ($T_{\text{min}}$) and the break point ($T_{\text{bp}}$) are indicated by vertical bars.

by several authors [12–14], [23] and is again confirmed here. The depression of $H_{c2}(T)$ at 6.0 K hence arises from the onset of the $q_{11}$ modulated magnetic structure. Table 1 adds a new correlation since $T_{H1}$ and $T_{\text{bp}}$ also nearly coincide. This strongly suggests that the onset of the helical structure with $q_{11} = 0.905c^*$ is responsible for the depression of $H_{c2}(T)$ at $T_{\text{bp}} = 5.7$ K for HoNi$_2$B$_2$C. It may be mentioned that anomalies in the critical current [11] and point contact spectroscopy [25] data of HoNi$_2$B$_2$C have been noticed at $T = 5.6$–$5.7$ K, without being ascribed to any change in the magnetic structure. A correlation between $T_{\text{min}}$ and $T_N$ can also be noticed for HoNi$_2$B$_2$C. On the one hand, this correlation is considered fortuitous by Amici–Thalmeier–Fulde in [18] since the $q_N$ Néel structure is considered to be strongly pair-breaking as the other AF structures: $H_{c2}(T)$ would recover at $T_{\text{min}} = T_N$ simply because the superconducting order parameter keeps on increasing, while the staggered AF magnetization saturates. On the other hand, the coincidence between $T_{\text{min}}$ and $T_N$ is considered to be a key feature in [15] because the non-nested $q_N$ Néel structure is considered to be less pair-breaking than the nested $q_M$ modulated structure, owing to the Machida–Ramakrishnan pair-breaking mechanism [16, 17]. Our data cannot help us to solve this question precisely but definitely indicate that both the nested $q_M$ modulated structure and the non-nested $q_{11}$ helical structure depress the superconducting order parameter more strongly than does the pair-breaking of the paramagnetic phase. Similarly to the specific heat data, no obvious feature in $H_{c2}(T)$ can be associated with the other $q_{12}$ helical structure, neither in our data nor in the literature. This is due to the absence of an abrupt onset for this AF structure [2–4], [5]. Based on the fact that the $q_{11}$ structure is pair-breaking, we propose that the $q_{12}$ magnetic order also continuously and smoothly depresses superconductivity between $\sim 8$ K and $T_N = 5.2$ K. From their magnetic x-ray scattering results (coherent scattering domain size), Hill et al [5] concluded that for $T_N < T \leq T_{H1}$ the $q_M$, $q_{11}$ and $q_{12}$ AF structures of HoNi$_2$B$_2$C coexist in spatially separate magnetic domains. Based on this scenario of coexistence of the three incommensurate magnetic structures (see the neutron part), we believe that above $T_N$, $H_{c2}(T)$ is a composite
curve resulting from the slightly differing magnetic properties of each of the spatially separate magnetic domains constituting HoNi$_2$B$_2$C. Paradoxically, this complex phenomenology makes the development of a comprehensive theory difficult, which is nonetheless crucially needed.

4. Conclusion

To summarize, we have been able to correlate the multiple anomalies seen in the specific heat data of HoNi$_2$B$_2$C to the details of the magnetic structures inferred from our neutron measurements: in particular, the peak at $T_{H1} = 5.7$ K arises from the onset of the $q_{H1} = 0.905c^*$ magnetic helical structure. We confirm that the decrease in $H_{c2}(T)$ for HoNi$_2$B$_2$C first seen at 6.1 K arises from the $q_M = 0.585a^*$ modulated magnetic structure, while another decrease seen at 5.7 K is attributed to the $q_{H1} = 0.905c^*$ magnetic helical structure. Both these antiferromagnetic structures are pair-breaking.

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