Lattice anisotropy in uranium ternary compounds

UTX


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Lattice anisotropy in uranium ternary compounds: UTX

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Several U-based intermetallic compounds (UCoGe, UNiGe with the TiNiSi structure type and UNiAl with the ZrNiAl structure type) and their hydrides were studied from the point of view of compressibility and thermal expansion. Confronted with existing data for the compounds with the ZrNiAl structure type a common pattern emerges. The direction of the U–U bonds with participation of the 5f states is distinctly the “soft” crystallographic direction, exhibiting also the highest coefficient of linear thermal expansion. The finding leads to an apparent paradox: the closer the U atoms are together in a particular direction the better they can be additionally compressed together by applied hydrostatic pressure.

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1. Introduction

One of the most striking features of the light-actinide compounds is a giant magnetic anisotropy, originating from sizeable orbital magnetic moments induced in the system of bonding itinerant electrons by a strong spin–orbit interaction. Such a situation leads to the so-called hybridization-induced anisotropy, which is two-ion by nature, and is therefore qualitatively different from a single-ion anisotropy due to CEF phenomena [1,2]. This bonding directionality manifests itself mainly at low occupancy of the f-states; more f-electrons lead to a wider directional spread of occupied m f states. U-systems with less than three 5f electrons per atom carry a hallmark of such phenomena in the orientation of 5f moments perpendicular to the shortest U–U bonds. It is most apparent in the case of uniaxial anisotropy, arising for a planar coordination of U, as in the ternary UTX compounds with the ZrNiAl structure type [3]. If U atoms are coordinated in linear chains (TiNiSi or GaGeLi structure types from the UTX compounds), a hard-magnetization axis is equivalent to the chain direction, with in-plane anisotropy perpendicular to the chain direction being weaker [3]. In this sense, the magnetic anisotropy reflects the bonding anisotropy in U-based compounds.

The UTX compounds with the ZrNiAl structure type were also found to exhibit a large elastic anisotropy [4]. Applying a hydrostatic pressure, the compression along the hexagonal basal-plane sheets (with shorter U–U spacing) is much higher than that along the c-axis. Such an effect observed for all studied U-based compounds and absent in rare-earth analogues indicates that the 5f states and 5f–5f bonds are the main culprit. It can be related to the sensitivity of the 5f states with highly variable degrees of delocalization to all control parameters. Ref. [4] also showed that the same pattern as the difference in linear compressibilities is followed by thermal expansion; the “soft” lattice directions also have the highest coefficients of linear thermal expansion. Such an elastic anisotropy has not yet been studied in other structure types, so its general validity (for U compounds) is uncertain.

Here we present results of high-pressure and thermal-expansion studies of several types of UTX compounds and their hydrides, which were selected to test a more general validity of the above mentioned rules, pointing out the specifics of light actinides. The crystallographic parameters of all investigated and some referenced UTX compounds are presented in Table 1. Hydrogenation is used as a tool to achieve a volume expansion. One has to be, however, cautious to interpret it as a negative pressure. The expansion due to the H absorption can be, as we observe, also highly
anisotropic, but the anisotropy can sometimes be different from that obtained by a hydrostatic pressure.

2. Experimental details

Polycrystalline samples of UNiAl, UNiGe, UCoGe, and UIrGe were prepared by arc melting of stoichiometric amounts of the constituent metals. The crystal structure was checked by means of XRD-3003 Seifert diffractometer (Cu-Kα radiation). In the case of UNiGe a certain amount (7–8%) of spurious phase (UNi₂Ge₂) was detected.

After initial preparation, hydrides of these compounds were synthesized. Prior to exposing a bulk material to hydrogen/deuterium, it was crushed into sub-millimeter particles and placed into a reactor. The surface of the sample was activated by heating up to T = 523 K under high-vacuum conditions (pressure of 10⁻⁶ mbar). Subsequently the activated material was exposed to a hydrogen (or deuterium) atmosphere. Depending on its pressure, various H/D stoichiometries can be achieved. The last step was thermal cycling of the sample up to T = 773 K at a given hydrogen/deuterium pressure to promote the absorption. The absolute amount of absorbed H/D can be determined by thermally induced desorption into an evacuated calibrated volume.

The temperature dependence of the lattice parameters of UNiAl₁₅, UNiGe, UNiGe and one of its hydrides (UNiGe₄₁) were studied using an Siemens-D5000 diffractometer (Co-Kα radiation). The X-ray diffraction (XRD) data were collected at ambient pressure and temperature decreasing gradually down to 5 K (or down to 80 K for UNiAl₁₅). Low-T diffraction data for UNiAl₁₅ were collected by means of the time of flight method at LANSCE LANL.

The high-pressure XRD experiments were performed at room temperature up to 22 GPa using a modified Bruker D8 diffractometer with focusing mirror optics, installed on a Mo rotating anode source (λ = 0.70926 Å). The micropowder of UNiAl₁₅ was placed in a diamond anvil cell (DAC) into the gasket (in our case mainly Be). High-pressure XRD data were collected on a Bruker Smart APEX II detector. Pressure was determined by means of the ruby fluorescence method and silicone oil was used as the pressure-transmitting medium. The diffraction images were processed using ESRF Fit2D software. The crystal structure refinements were done using the FullProf software suite.

3. Experimental results and discussion

3.1. Switching over the soft direction in the ZrNiAl family

The hexagonal ZrNiAl-type of structure (space group P-6m2), which is the ordered ternary variant of the Fe₃P-type, has a distinct layered character. It is built up of U-T and T-X basal-plane layers alternating along the c-axis. The shortest U-U distances (d₁,U-U) are found within the U-T layers, where each U-atom has four nearest U neighbours. The U-T layers are separated by the lattice parameter c. Hydrides/deuterides of hexagonal UTX usually adopt the same type of structure as their precursors, but exhibit a large volume expansion. UNiAl was the first such compound, in which a large hydrogen absorption was detected, which leads to a large expansion in the basal plane, and a small compression in the c-direction [5]. More detailed structure studies [6,7] indicated that the U-U spacing within the basal plane increases enormously (d₁,U-U = 3.48 Å in UNiAl and d₁,U-U ≈ 4.15 Å in its deuteride) so that it becomes higher than the c-axis spacing (3.98 Å). This would lead, provided the two-ion anisotropy model is correct, to a swapping of the anisotropy type from c-axis to the basal plane. This assumption could not be corroborated by neutron diffraction, as the magnetic structure could not be resolved from a small amount of magnetic reflections [8]. Neither single-crystal could be synthesized to determine the anisotropy of bulk susceptibility.

Therefore we undertook the high-pressure crystal-structure study of UNiAl₂₁ with the aim to find a possible change of the soft crystallographic direction from the a-axis (basal plane) to the c-axis direction. Results of the room temperature XRD study under applied pressure up to 22 GPa are displayed in Fig. 1, together with similar data for UCoAl from Ref. [4]. The high-pressure XRD experiment was not performed for pure UNiAl, but because of the uniformity of behaviour of U compounds with the same structure type [4] one can expect close similarity of UNiAl and UCoAl.

The experiment was performed with gradually increasing pressure. After reaching the maximum pressure, a few data points were taken also during unloading. The agreement of lattice parameters of initial and final ambient-pressure state excludes that the H concentration would change during the high-pressure experiment. The pressure variations of individual lattice parameters and volume were fitted to a quadratic polynomial dependence (p = g₁𝑝₀ − k₂𝑝₀ + k₃𝑝₀²) which yielded the linear compressibility k₁ each along lattice direction i (Table 2). The bulk modulus can be expressed as B₀ = 1/|kᵥ|, where kᵥ is the volume

![Fig. 1. Pressure variations of the relative change of the lattice parameters a and c for UNiAl₂₁ compared with UCoAl [4].](image-url)
compressibility, which can be obtained from the fit of $V(p)$, or from the sum of individual compressibilities along given lattice directions ($k_v = 2k_a + k_c$).

The respective data reveal that the $a$-axis compressibility of UNiAlD$_{2.1}$ is indeed somewhat lower than that in UCoAl (see Fig. 1). Even much more striking is the difference for the $c$-axis compressibility. The factor of almost 10 underlines the trend observed. The almost incompressible $c$-axis direction becomes the soft one in the deuteride by bringing the U atoms closer together in this particular direction, and pulling them apart in other two directions. The bulk modulus for UNiAlD$_{2.1}$ ($B_0 \approx 127$ GPa) is in the range of the typical values observed for UTX compounds with the ZrNiAl structure type [4].

3.2. Effect of pressure in UTX compounds with the TiNiSi structure type

UTX compounds with X = Si or Ge tend to form in the structure of the TiNiSi-type. Here, it may be the soft orthorhombic $a$-direction, following the basic U–U coordination, forming U–U zig–zag chains stretched along the $a$-axis. Indeed, the U magnetic moments cannot be oriented along $a$-direction in UNiGe [3], UCoGe [9] or UIrGe [10] even under influence of high magnetic fields, in analogy with the situation within the ZrNiAl-structure family, where the moments cannot be rotated out of the $c$-axis.

Hydrogenation of such compounds leads to a more symmetric hexagonal structure (ZrBeSi-type of structure), with the U chains stretched and aligned along the direction, which becomes now the hexagonal axis $c$ [11]. Following a traditional notation we call these the $\beta$-hydrides. Besides those, $\alpha$-hydrides with a much smaller H concentration, which preserve the original lattice and only slightly expand it, could be prepared in several cases.

It was tempting to investigate whether the bonding anisotropy analogous to ZrNiAl structure type exists also for the TiNiSi structure type and derived hydrides. The structural relations between the three mentioned types of structures (ZrNiAl-, TiNiSi- and ZrBeSi-type) are shown in Fig. 2. The most interesting case is the weakly ferromagnetic unconventional superconductor UCoGe, which rapidly loses ferromagnetism with applied pressure [12], but also in the state of $\alpha$-hydride, which has slightly higher unit-cell volume [13].

The results of the high-pressure study of UCoGe performed in analogy to the experiment on UNiAlD$_{2.1}$ are summarized in Fig. 3 and Table 2. The $a$-direction is about an order of magnitude more compressible than the $b$- and $c$-directions. The ratio of the respective linear compressibilities is very similar to the UTX compounds with the ZrNiAl structure type. The difference is the multiplicity of the “soft” direction. Two soft and one hard direction in the ZrNiAl structure is replaced by one soft and two hard directions in the TiNiSi structure type. As the compressibility values for the respective hard and soft directions are quite similar, the bulk modulus must be considerably higher for the TiNiSi structure. The value of the obtained bulk modulus of UCoGe ($B_0 = 309$ GPa) [14] is indeed the highest one reported amongst the UTX family, but it was in line with results obtained on UTSi-H compounds, forming in a structure similar to TiNiSi ($B_0 = 257$ GPa for UPdSi$_{1.0}$) [15].

As shown above, hydrogenation is a convenient tool to modify the crystal lattice. It acts primarily as the negative pressure agent resulting in the volume expansion of the crystal lattice. Details show, however, that the expansion is anisotropic (Fig. 4), and the anisotropy can be different than at the hydrostatic compression. It is instructive to make the comparison on the case of UCoGe. The lattice variations between UCoGe and its $\beta$-hydrides show the same anisotropy as a hypothetical negative pressure. As the $\beta$-hydride of UCoGe becomes hexagonal and the U chains along a stretch and form a straight line, the $a$-axis (keeping the orthorhombic notation, i.e. the $c$-axis in the hexagonal notation) consequently expands significantly, and the $b$- and $c$-axis shrink. Such compounds exhibit a stronger ferromagnetism than pure UCoGe [13]. However the $\alpha$-hydride, seen in the inset of Fig. 4, reveals a different pattern. For the H concentrations around 0.1H/f.u. it is the $c$-axis which

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**Table 2**

Overview of linear compressibilities $k_a, k_b, k_c$, their derivatives and bulk moduli $B_0$ and their derivatives.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Linear compressibilities</th>
<th>Isothermal bulk modulus</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$a$-axis</td>
<td>$b$-axis</td>
</tr>
<tr>
<td>UCoGe$^a$</td>
<td>2.8</td>
<td></td>
</tr>
<tr>
<td>UNiAl$^a$</td>
<td>2.8</td>
<td>13</td>
</tr>
<tr>
<td>UNiAlD$_{2.1}$</td>
<td>2.1</td>
<td></td>
</tr>
<tr>
<td>UIrGe</td>
<td>1.55</td>
<td></td>
</tr>
<tr>
<td>UCoGe$^b$</td>
<td>2.3</td>
<td>0.6</td>
</tr>
</tbody>
</table>

* $^a$ [4].
* $^b$ [14].

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![Fig. 2](image1.png) **Fig. 2.** Scheme showing approximate correspondence of individual crystallographic directions in the three structure types discussed in the text.

![Fig. 3](image2.png) **Fig. 3.** Pressure variations of the relative change of lattice parameters $a$, $b$, $c$ and volume $V$ for UCoGe.
expands most, whereas b is decreasing and a staying approximately constant. This is very different than for a negative pressure, and therefore it is not that surprising that the weak ferromagnetism vanishes, despite the volume expansion (0.25%), similarly to the positive pressure influence [13]. The $d_{ij}(U)$ values stay approx. constant, about 3.48 Å. By stretching the U-chains, the intra-chain U–U spacing increases up to 3.97 Å and the inter-chain U–U spacing up to 4.10 Å in the $\beta$-hydride with the volume expansion of 10.7%.

No $\alpha$-hydride could be prepared for UNiGe, which is similar to the situation in UNiAl. Three samples of $\beta$-hydrides of UNiGe with different H content (0.3, 1.0 and 1.21H/f.u.) were prepared using various H2 pressures. The compound becomes a ferromagnet with $T_C$ approaching 100 K for the highest H concentration [16]. An inspection of the XRD patterns reveals that all crystallize in the hexagonal ZrBeSi-type of structure. An attempt to obtain an $\alpha$-hydride resulted in a mixture of two phases (non-expanded orthorhombic and hexagonal ones). The formation of $\beta$-hydrides from UNiGe is accompanied by a strongly anisotropic lattice change as it can be seen e.g. for UNiGeH1.0 ($\Delta/a = 10.1\%$, $\Delta/b = -3.1\%$, $\Delta/c = -1.2\%$) with the volume expansion of 5.8%, in the representation of orthorhombic unit cell. Clearly, the relative lattice expansion/compression along a and b directions follows the same pattern as that reported for UCoGe or e.g. for LaNiSn hydrides ($\Delta/a = 12.9\%$, $\Delta/b = -5.1\%$, $\Delta/c = 0.7\%$) with volume expanded by about 7.9% [17]. The only difference in the trend is observed along the c-direction. Both orthorhombic lattice parameters b and c are reduced in the UNiGe hydrides with respect to the original value. The inter-U spacing increases consequently, e.g. from $\approx3.56$ to 3.86 Å in UNiGeH1.0. This is still smaller than the inter-chain distance of 4.10 Å. For this compound, as well as for several other TiNiSi-type compounds (hydrogen-free), we studied the thermal expansion by XRD and compared it with the data for the ZrNiAl structure family. The results are given in the next section.

3.3. Thermal expansion of UTX compounds – anomalous case of URuGa

The thermal expansion of all investigated compounds crystallizing in various structure types is highly anisotropic. This reflects both the anisotropy seen at elevated temperatures, which can be assumed as the lattice effect, and the anisotropy associated with magnetic ordering, observable at temperatures comparable with respective ordering temperatures and below. The lattice effect can be parameterized by the linear thermal expansion coefficient $\alpha$, taken from the linear high-temperature part as $d$($\Delta$/L)/dT. $N$ coefficients $\alpha_i$ are related to compressibilities $k_i$ as follows:

$$\alpha_i = k_i \Gamma_{0i}C_V/V,$$

where $C_V$ is the specific heat at constant volume, V is the molar volume, and $\Gamma_{0i}$ is the Grüneisen parameter, which can be very anisotropic itself [4]. The low-temperature effects can be related to the spontaneous magnetostriction, commonly seen as a sizeable effect in band magnets [18]. The volume increase due to formation of magnetic moments is essentially related to a slight increase of kinetic energy of the electrons due to the splitting of the sub-bands. The anisotropic lattices (and related anisotropic Fermi surfaces) of the UTX compounds demonstrate that even such an effect can be highly anisotropic, with a variable sign of the lattice.

Fig. 4. Relative changes of lattice parameters $a$, $b$, $c$ and volume V for UCoGe as a function of H concentration. The lattice parameters of hexagonal hydrides of UCoGe were recalculated for the orthorhombic representation.

Fig. 5. Temperature dependence of the relative expansion along $a$, $b$, and $c$-axes for different structure types. The relative volume changes are represented by the red lines. The relations between the lattice parameters of TiNiSi- and ZrBeSi-types of structure are expressed by corresponding colours. Large circles for UNiAl, express the results of neutron diffraction experiment. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)
reaction to magnetism. The mutual cancellation can give a more modest volume effect.

Concerning the compounds with the ZrNiAl structure type, the magnetostriction typically leads to expansions in the c-direction and compression in the a-direction. It is quite general, as observed in the ferromagnet: UPTAI [19] and antiferromagnets: UNiAI [20] or UNiGa [21]. The size of the magnetostriction effect is however several times larger in the UNiAl deuteride, where it reaches the order of $10^{-3}$. Apparently the opposite tendency was observed with URuGa, showing a pronounced anomaly developing below $T = 120\text{ K}$ and reaching more than $1 \times 10^{-3}$ for linear expansions [22]. It is, however, not clear for this compound whether the anomalies are related to magnetism. URuGa is a spin fluctuator with a characteristic temperature, below which magnetic susceptibility flattens off, of approx. 80 K [22,23]. On the other hand, lattice properties of URuGa can be perceived as anomalous amongst the U-based ZrNiAl-type of compounds. The anomalously high lattice parameter $a = 7.115(3)$ Å and small $c = 3.836(2)$ Å lead to the $d_{0.1}$ value in the basal plane of 3.73 Å which is only marginally smaller than the c-axis spacing of 3.836(2) Å [23]. Considering the anomalies in the temperature dependence ($a$, $c$ ($T$)) of the lattice parameters $a$ and $c$, we can estimate the low-temperature $d_{0.1} = 3.723$ Å for $a$ and 3.823 Å for $c$, which brings the values even closer together. It represents therefore a bridge between UNiAlD$_{2.1}$ and the rest of the U-compounds with this structure type. We undertook to test the situation of URuGa under hydrostatic pressure. The difference of linear compressibilities is rather indistinct, although $k_{a} = 1.55 \times 10^{-3}$ GPa$^{-1}$ is still somewhat higher than $k_{c} = 1.19 \times 10^{-3}$ GPa$^{-1}$. The high-temperature linear thermal expansion is then in-line with expectations being almost isotropic with both directions yielding approx. $11 \times 10^{-3}$ K$^{-1}$ [22].

The temperature dependence of the relative changes of lattice parameters and the volume for the deuteride of UNiAl is shown in Fig. 5. The magnetostriction type corresponds to the parent compound [24], with expansion along $c$ and compression along $a$ below the phase transition seen around 100 K. The lattice thermal expansion at room temperature is extremely anisotropic, with the c-axis expansion about 50 times higher than that along $a$-axis. The type of anisotropy corresponds to the compressibility anisotropy, with the soft direction $c$. The reversal of the more expanding direction comparing to other U compounds with the ZrNiAl structure may be again directly related to the reversal of the U–U coordination.

An interesting opportunity is offered by the analogy with the compounds of the TiNiSi type. The hexagonal basal plane of the ZrNiAl-type of structure is in some respect equivalent to the $a$–$c$ plane of the TiNiSi-type structure [25]. But the shortest $U$–$U$ spacings are within the chains stretched along the $a$-axis, and indeed the highest thermal expansion was found along $a$ (see Table 3). It corresponds to the most compressible direction (observed for UCoGe), which is also the hard-magnetization direction for UNiGe [3] and also for UCoGe [9]. The least expanding $b$-axis corresponds to the least compressible direction in UCoGe.

Although standard XRD is not a tool capable of distinguishing magnetostriction phenomena on the level of $10^{-4}$ relative expansion, one can recognize anomalies associated with magnetic ordering, which setting in at 50 K with the propagation vector $q = (0, 0.359, 0.359)$ [26,27]. The $b$-axis (having the role of $c$-axis for e.g. UNiAl) follows the tendency of the $c$-axis in UNiAl [15], revealing a noticeable increase in the ordered state, reaching approx. $\Delta b/b = 10^{-3}$. Anomalies on other two axes are weaker.

The isostroic compound UGeGe behaves somewhat differently. The soft direction is again the $a$-direction, however the essential difference is observed along the $b$-axis. Interestingly, UGeGe is also resistant to high $H_2$ pressure. We succeeded to obtain only an $\alpha$-hydride (UGeGeH$_{2.1}$) at the highest available $H_2$ pressure of 140 bar, no $\beta$-hydrides were formed under such conditions. The crystal structure remained orthorhombic with the volume expansion of 0.7%. The lattice expands anisotropically, mainly along the $c$-axis by about 0.43% and 0.20% along $a$-axis, no expansion is observed in the $b$-direction. UGeGeH$_{2.1}$ orders ferromagnetically below 28 K, although the parent compound is an antiferromagnet below 16–17 K. This magnetic phase transition for UGeGe is also reflected by a small anomaly in the $c$-direction around this temperature. For the hydride of UNiGe, the $c$-direction is the softer one, which is in agreement with pure UNiGe, as the lattice parameter $c_{\text{hex}}$ corresponds to $a_{\text{trid}}$.

### 4. Summary and conclusions

The examples collected from representative U compounds of two different structure types, namely ZrNiAl and TiNiSi, demonstrate that the lattice properties, despite the large differences in e.g. bulk modulus, follow the same pattern of anisotropy. The direction with the closest U–U separation is the soft (in terms of compressibility) crystallographic direction, and this direction has also a higher coefficient of thermal expansion at high temperatures, where the lattice contribution to thermal expansion dominates. The results imply that a room-temperature study of compressibility or thermal expansion can provide information on magnetic structure, which realizes at low temperatures. The study indicates that attempts to scale variations of bulk properties to applied hydrostatic pressure (e.g. around the quantum critical point) may be actually rather misleading, and one has to be concerned with pressure variations of individual lattice parameters. This is true for the hydrogenation, as well.

The test case UNiAlD$_{2.1}$, which is an exception from the planar coordination of U atoms in the ZrNiAl structure type, has a special importance. It indicates that a reversal of the soft direction happens following the reversal of the U–U coordination within the same structure type, which is induced by an anisotropic expansion upon the H absorption.

The fact that the 5f electronic states tend to bond only to the nearest U neighbours forming a “soft” bond leads to an apparent
paradox – the closer the U atoms are together, the easier they can still be pushed together by hydrostatic pressure. Such a situation can naturally occur only at a moderate U–U spacing. For very large U–U spacing in compounds with low U concentration the directionality of U–U bonds (even if it can be mediated by the Sf-ligand hybridization) should vanish, and it would be very interesting to follow the variations of elastic anisotropy into such systems. On the other hand we can also expect weakening of elastic anisotropy in close-packed structures.

As a conclusion, we have demonstrated that the observed anisotropy of elastic properties of U intermetallic compounds is due to the directionality of the U–U bonding.

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