



# Influence of competing energy scales on the effective spin degeneracy in $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$

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## ABSTRACT

The interplay between Kondo effect, RKKY interaction and crystal field (CF) splitting in the pseudo-ternary substitution series  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  ( $0 \leq x \leq 1$ ) is investigated by means of X-ray powder diffractometry and specific heat studies. Contrary to  $\text{CeNi}_8\text{CuGe}_4$  and  $\text{CeNi}_8\text{CoGe}_4$  no long range magnetic order occurs in  $\text{CeNi}_8\text{FeGe}_4$  since it basically retains a four-fold degenerate Kondo lattice ground state similar to its parent system  $\text{CeNi}_9\text{Ge}_4$ . Kondo screening as well as a modest CF splitting between the two lower energy states ( $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$ ) increase simultaneously with increasing Fe concentration resulting in a Kondo dominated ground state for the whole substitution region. A new parameter  $\alpha = T_{R\ln 4}/T_{R\ln 2}$  is introduced to analyze the interplay between the competing energy scales.

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## 1. Introduction

In the last years a rich variety of different ground states has been discovered in the Ce based intermetallic systems  $\text{CeT}_9\text{X}_4$  (T: transition metal, X: group 14 element) [1] such as: (i) intermediate valence behavior in  $\text{CeCo}_9\text{Si}_4$  [2], (ii) Kondo lattice behavior in  $\text{CeNi}_9\text{Si}_4$  [3] and (iii) long range magnetic ordering in  $\text{CeCo}_9\text{Ge}_4$  [4].

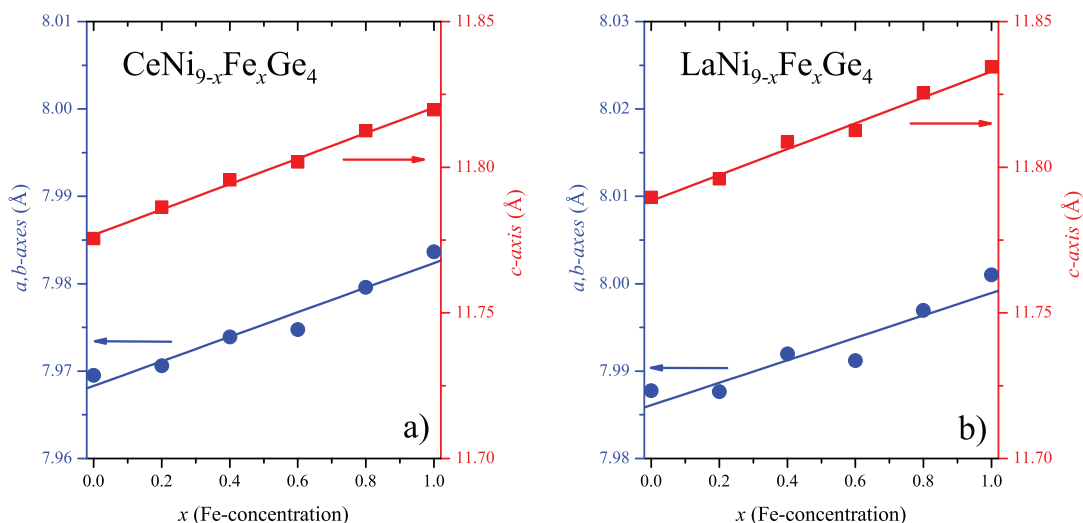
The most outstanding compound is, however, the heavy fermion system  $\text{CeNi}_9\text{Ge}_4$  which is positioned at the borderline between Fermi-liquid and non-Fermi-liquid physics [5,6]. The so far highest recorded Sommerfeld coefficient  $\gamma$  of about  $5.8 \text{ J/mol K}^2$  for paramagnetic Ce Kondo-lattices is mainly due to a single ion effect solely based on local magnetic fluctuations [6,7]. The Hund's rule ground state of  $\text{Ce}^{3+}$  with  $J=5/2$  splits in the tetragonal crystal field, generated by the square-antiprismatic environment, into three Kramer's doublets. The two lowest doublets ( $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$ ) reveal a minute energetic separation of  $\Delta_1 \simeq 6 \text{ K}$  and are energetically well separated from the higher lying  $\Gamma_6$  doublet ( $\Delta_2 \simeq 128 \text{ K}$ ) [8]. Due to the fact that the Kondo energy  $T_K = 3 \text{ K}$  is of the same order of magnitude as  $\Delta_1$ , the first excited doublet  $\Gamma_7^{(2)}$  contributes

significantly to the Kondo ground state. Hence the total magnetic response differs from a simple  $SU(N)$ -Anderson impurity model, and has been described by a crossover scenario from  $SU(2)$  to  $SU(4)$  [9,10]. As the splitting of the two Kondo broadened  $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$  doublets of  $\text{CeNi}_9\text{Ge}_4$  is neither resolved in specific heat nor by cold neutron inelastic scattering we denote the crystal field ground state as a quasi-quartet (effectively four-fold degenerate). Coleman [11] proposed theoretically that at a given Kondo interaction strength a system with larger orbital degeneracy would be less likely to order magnetically. Indeed, no magnetic order is observed in  $\text{CeNi}_9\text{Ge}_4$  down to the lowest experimentally reached temperatures (35 mK).

Subsequent substitution of Ni by the neighboring transition metals, Cu and Co, causes in both cases a reduction of the effectively four-fold degeneracy of the Kondo ground state toward an effectively two-fold one and long range antiferromagnetic (AF) order is observed with  $T_N = 0.2 \text{ K}$  and  $1 \text{ K}$  for  $\text{CeNi}_8\text{CuGe}_4$  and  $\text{CeNi}_8\text{CoGe}_4$ , respectively [12,13]. In case of Cu doping a tendency toward AF order is indeed expected due to an increase of the 3d-electron count. Whereas, in the case of Co admixture the formal reduction of the 3d-electron count surprisingly results in an AF transition as well. The latter observation is counter-intuitive in the framework of the standard Doniach picture [14]. These results clearly suggest that it is rather the reduction of the effective spin degeneracy  $N$  [12,15] away from the paramagnetic quasi-quartet ground state in  $\text{CeNi}_9\text{Ge}_4$  which controls the formation of AF order in both systems.

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**Fig. 1.** The tetragonal lattice parameters  $a=b$  and  $c$  of annealed samples (a)  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  and (b)  $\text{LaNi}_{9-x}\text{Fe}_x\text{Ge}_4$  with respect to the Fe concentration  $x$ . The solid lines are linear fits following Vegard's law.

To gain further insight, whether (i) the interplay between Kondo effect and RKKY interaction or (ii) a change of the effective spin degeneracy is the most relevant control parameter in fine-tuning the  $\text{CeNi}_{9-x}\text{T}_x\text{Ge}_4$  systems (T: Co, Cu), we performed specific heat studies of  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  ( $x \leq 1$ ). This solid-solution series allows a further depletion of the  $3d$ -electron count in comparison to the respective Co series.

## 2. Experimental techniques

X-ray powder diffraction studies using  $\text{Cu K}\alpha$  radiation ( $\lambda = 1.54178 \text{ \AA}$ ) were employed to identify the crystal structure of the  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  phases and to monitor their purity. Additional energy dispersive X-ray (EDX) analysis and inductively coupled plasma (ICP) measurements were used to determine the samples' stoichiometry and to trace potential impurities. Specific heat experiments were performed with a commercial physical property measurement system (PPMS) between 2 K and 300 K. For temperatures between 0.06 K and 2.8 K a self designed specific heat device using a standard relaxation method [16] was installed in a  $^3\text{He}/^4\text{He}$ -dilution refrigerator.

## 3. Sample preparation and structural characterization

Polycrystalline samples  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  and  $\text{LaNi}_{9-x}\text{Fe}_x\text{Ge}_4$  ( $x = 0, 0.2, 0.4, 0.6, 0.8, 1$ ) were prepared by arc-melting stoichiometric amounts of the respective elements as explained for the Co system elsewhere [4]. The final ingots were characterized by small traces of the pseudo-binary ferromagnetic  $\text{Ni}_{3-x}\text{Fe}_x\text{Ge}$  impurity phase ( $x_i = 3 \pm 1 \text{ wt.}\%$ ), which are however insignificant for the interpretation of specific heat studies. Energy dispersive X-ray analysis and inductively coupled plasma measurements reveal for  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  an error in the  $x$  stoichiometry parameter of less than 3.5%. Rietveld refinements of X-ray powder diffraction data are in line with the tetragonal  $\text{LaFe}_9\text{Si}_4$  [17] (space group  $I4/mcm$ ) structure type in case of all solid solutions of  $\text{Ce/LaNi}_{9-x}\text{Fe}_x\text{Ge}_4$  ( $0 \leq x \leq 1$ ).

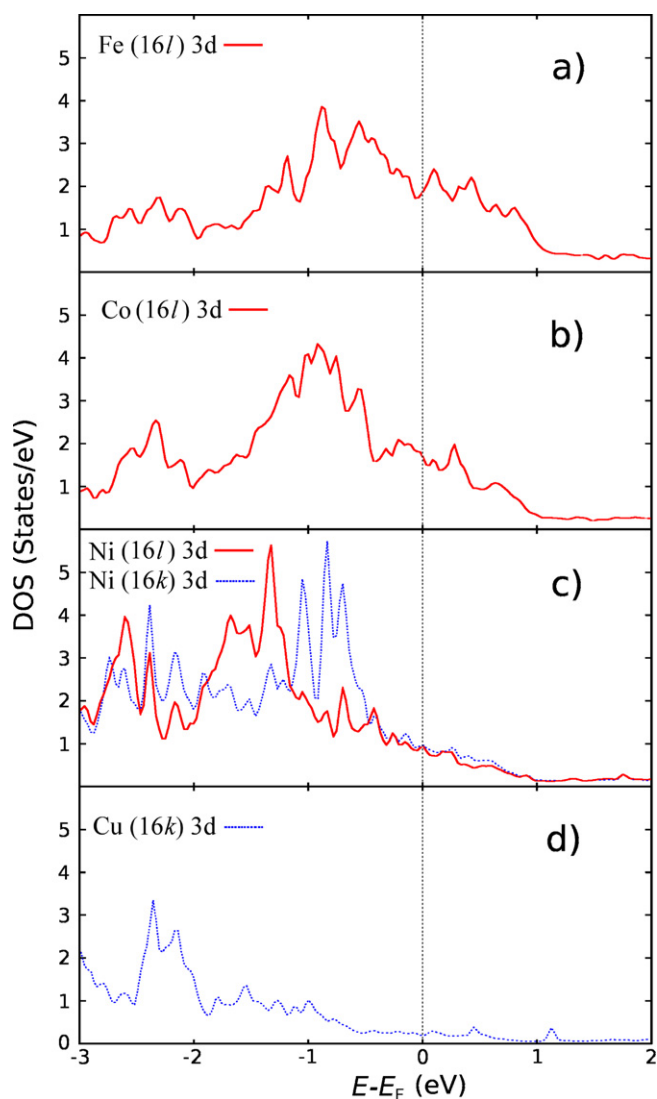
This analysis reveals that stepwise nickel by iron replacement is accompanied by a slight volume expansion (maximal 0.7%) for  $x=1$  for both, the cerium and the corresponding lanthanum system. As shown in Fig. 1 the concentration dependence of the tetragonal lattice parameters  $a=b$  and  $c$  follows Vegard's law for both solid solutions  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  and  $\text{LaNi}_{9-x}\text{Fe}_x\text{Ge}_4$ . This observation is different to the Co substitution series where no significant volume change was observed in the stoichiometric range of  $0 \leq x \leq 1$  [13]. On contrast, the clear change of chemical pressure in the  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  series upon Ni by Fe substitution should

significantly influence the hierarchy of competing Kondo, RKKY and CF interactions and help to identify the control parameter for the onset of AF ordering at low temperatures.

## 4. Electronic structure calculations

The parent compound  $\text{CeNi}_9\text{Ge}_4$  (space group  $I4/mcm$ ) crystallizes in the tetragonal  $\text{LaFe}_9\text{Si}_4$  structure type [5]. Here, the rare earth elements Ce and La occupy the crystallographic  $4a$  sites of 422 point group symmetry. In the case of solid solutions  $\text{CeNi}_8\text{TGe}_4$  the main group element Ge is situated on the  $16l$  sites and the transition metal elements T are distributed over the  $16k$ ,  $16l$  and  $4d$  sites. In order to investigate a possible site-preference for the substitution of Fe on one of the three crystallographically inequivalent Ni sites of  $\text{CeNi}_9\text{Ge}_4$  DFT electronic structure calculations employing the WIEN2K suite of programs have been carried out [18]. Previous theoretical studies on the substitution of nickel by copper predicted, that the  $16k$  sites ( $m..$  point symmetry) are energetically preferred over the  $16l$  and  $4d$  sites ( $..m$  and  $m..mm$  point symmetry, respectively) [12]. This result was later confirmed by neutron powder diffraction experiments and subsequent Rietveld refinements which revealed that "on average each fourth Ni atom at the  $16k$  sites is substituted by copper" [15]. In case of the  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  solid solution series, our calculations suggest that substitution occurs preferentially at the  $16l$  sites. Indeed, replacement of 25% of the Ni atoms by Fe at the  $16k$  and  $4d$  sites leads to an increase of the total energy by 0.2 and 0.1 eV/f.u. relative to the alternative substitution at the  $16l$  sites, respectively. Additional calculations on  $\text{CeNi}_8\text{CoGe}_4$  indicate the same site-preference for the  $16l$  position. In this system the increase of the total energy for the Ni by Co substitution at the  $16k$  and  $4d$  sites is found to be 0.1 eV/f.u. in both cases.

The observed site preferences might be correlated with the values found for  $N(E_F)$  of the site and state projected DOS of the T  $3d$  states: The substitution of Fe on the preferred  $16l$  site leads to a reduction of  $N(E_F)$  compared to the substitution on the  $16k$  site by 59%. In line with the smaller energetic preference of the  $16l$  site for T=Co this decrease of  $N(E_F)$  is also smaller and amounts to only 7%. In the case of T=Cu the substitution on the preferred  $16k$  site also leads to a  $N(E_F)$  value lowered by 12% relative to the alternative substitution on the  $16l$  site. Fig. 2 depicts the DOS of the T  $3d$  states at the preferred sites for T=Fe, Co, Cu. In the case of T=Ni the DOS of both sites ( $16l$  and  $16k$ ) (Fig. 2c) are depicted. With increasing  $d$  electron count of the T metal atoms the T  $3d$  states shift to lower



**Fig. 2.** Site- and state projected density of states (DOS) of the transition metal 3d states of  $\text{CeNi}_8\text{TGe}_4$  ( $T = \text{Fe, Co, Ni, Cu}$ ). The transition metal atoms are located at the energetically favored sites (Fe, Co 16l, Cu 16k). For  $T = \text{Ni}$  the 3d DOS of both crystallographically independent positions are shown.

energies relative to  $N(E_F)$ . As a consequence of this, the contributions of the 3d states to  $N(E_F)$  decrease along the row Fe, Co, Ni, Cu (2.97, 1.82, 1.05 and 0.17 states/eV, respectively).

## 5. Results and discussion

In Fig. 3 the specific heat divided by temperature  $C/T$  is displayed in the temperature region between 0.06 and 300 K for both substitution series  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  and  $\text{CeNi}_{9-x}\text{Co}_x\text{Ge}_4$  ( $0 \leq x \leq 1$ ). Besides the high temperature regime of the specific heat, which is dominated by phonon contributions,  $C/T$  of  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  exhibits a decrease of the Sommerfeld coefficient  $\gamma$  from approximately 5.8 J/mol K<sup>2</sup> for  $\text{CeNi}_9\text{Ge}_4$  to about 0.7 J/mol K<sup>2</sup> for  $\text{CeNi}_8\text{FeGe}_4$  at low temperatures (Fig. 3a). This enormous change of the  $\gamma(x)$  values in the low temperature regime signals in a rough estimation a growth of the Kondo energy ( $T_K \propto 1/\gamma$  [19]) with increasing  $x$ . For a more detailed analysis of the  $T_K$ -dependency the effective spin degeneracy has to be involved (see below).

Furthermore, there is no indication for any magnetic ordering as it occurs in the equivalent Co substitution series (Fig. 3b), where an antiferromagnetic transition sets in for  $x \geq 0.4$  [13]. The

observation of magnetic order in  $\text{CeNi}_{9-x}\text{Co}_x\text{Ge}_4$  is surprising because a reduction of the 3d electron count, accompanied with an increase of the  $N(E_F)$  (see Fig. 2), is expected to drive the balance between Kondo screening and RKKY interaction toward a Kondo dominated ground state [20].

To elucidate accurately the change of the individual energy scales which are relevant for the iron system, e.g., Kondo screening and CF splitting, we derived at first the magnetic contribution of the Ce sublattice by subtracting the total specific heat of the  $\text{LaNi}_{9-x}\text{Fe}_x\text{Ge}_4$  samples from the one of the corresponding  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  series. As a result we obtained the Ce based magnetic contribution  $\Delta C$  of  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  as depicted in a semilogarithmic plot in Fig. 4. The reliability of  $\Delta C$  is indicated by vertical error bars assuming a 1% uncertainty of the subtracted data. The error bars increase to high temperatures because of the relative large phonon contribution which also causes a significant error margin with respect to the position of the Schottky anomaly showing up at about 35 K.  $\Delta C$  of  $\text{CeNi}_9\text{Ge}_4$  is characterized by two pronounced maxima at about 5 K and 35 K. The lower one represents a Kondo contribution which is associated with a CF quasi-quartet ground state of the  $\text{Ce}^{3+}$ -ion ( $T_K \approx 3$  K and  $\Delta_{(\Gamma_7^{(1)}-\Gamma_7^{(2)})} \approx 6$  K) [8]. The upper Schottky-like maximum is associated with the presence of a  $\Gamma_6$  doublet at higher energy.

For  $\text{CeNi}_{8.8}\text{Fe}_{0.2}\text{Ge}_4$  the upper maximum increases and remains at the same temperature while the Kondo-type anomaly decreases as it is also observed in the homologous  $\text{CeNi}_{8.8}\text{T}_{0.2}\text{Ge}_4$  ( $T = \text{Co, Cu}$ ) [12,13]. This common behavior at lowest level of substitution is most likely a consequence of disorder introduced in the pseudo-ternary solid solution. The final trend of the substitution of Ni by Fe ( $x \geq 0.4$ ) is revealed by a decrease of the upper Schottky-like maximum which shifts to lower temperatures and by the reversed shift of the Kondo-like maximum to higher temperatures. For  $x \geq 0.8$  both maxima almost merge into one single feature at 9 K. For  $\text{CeNi}_8\text{FeGe}_4$  this pronounced maximum is in approximate agreement with the Coqblin–Schrieffer (CS) model [21] for a four-fold degenerate ( $N=4$ ) ground state using  $T_0 = 20$  K (solid line in the insert of Fig. 4). Some deviation is visible in the temperature range from about 20–60 K and is attributed to an additional Schottky contribution originating from the upper CF doublet  $\Gamma_6$ . The calculation of the characteristic temperature  $T_0 = (N-1)\pi R/6\gamma$  from the experimental value of the Sommerfeld coefficient  $\gamma \approx 0.7$  J/mol K<sup>2</sup> yields  $T_0 = 19$  K in close agreement with the above fit. The relation between  $T_0$  and  $T_K$  via the Wilson number  $W_J$  for  $J = 3/2$  [22] indicates for  $\text{CeNi}_8\text{FeGe}_4$  a Kondo temperature of  $T_K = W_J \cdot T_0 = 0.5843 \cdot T_0 \sim 12$  K.

The Kondo contribution to the specific heat of  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  with  $0.4 < x \leq 1$  is well described by the  $N=4$  CS-model. This indicates a ground state transformation from the  $SU(2)$  to  $SU(4)$  crossover scenario (in  $\text{CeNi}_9\text{Ge}_4$ ) to a well-defined  $N=4$  Kondo ground state.  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$ , thus, represents one of the rare examples where a  $N \leq 4$  to  $N=4$  ground state transformation can be experimentally enforced in a substitution series.

These assumptions are supported by the evolution of the temperature dependent magnetic entropy,  $\Delta S(T)$ , as displayed in Fig. 5 for  $\text{CeNi}_9\text{Ge}_4$  and  $\text{CeNi}_{8.6}\text{Fe}_{0.4}\text{Ge}_4$ . Entropy calculations of the parent compound  $\text{CeNi}_9\text{Ge}_4$  reveal a strong initial low temperature increase suggesting that the CF ground state of the  $\text{Ce}^{3+}$ -ion is generated by the two Kramer's doublets  $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$  [6,10], with a minute energetic separation of  $\Delta_1 \approx 6$  K. The shift of  $\Delta S(T)$  for  $\text{CeNi}_{8.6}\text{Fe}_{0.4}\text{Ge}_4$  to higher temperatures indicates a splitting of the quasi-quartet ground state into two single Kramer's doublets. The onset temperatures where the entropy approaches the  $R \ln 2$  and  $R \ln 4$  limit are plotted in the insert of Fig. 5 versus the Fe concentration  $x$ . The rise of  $T_{R \ln 2}$  and  $T_{R \ln 4}$  with increasing concentration

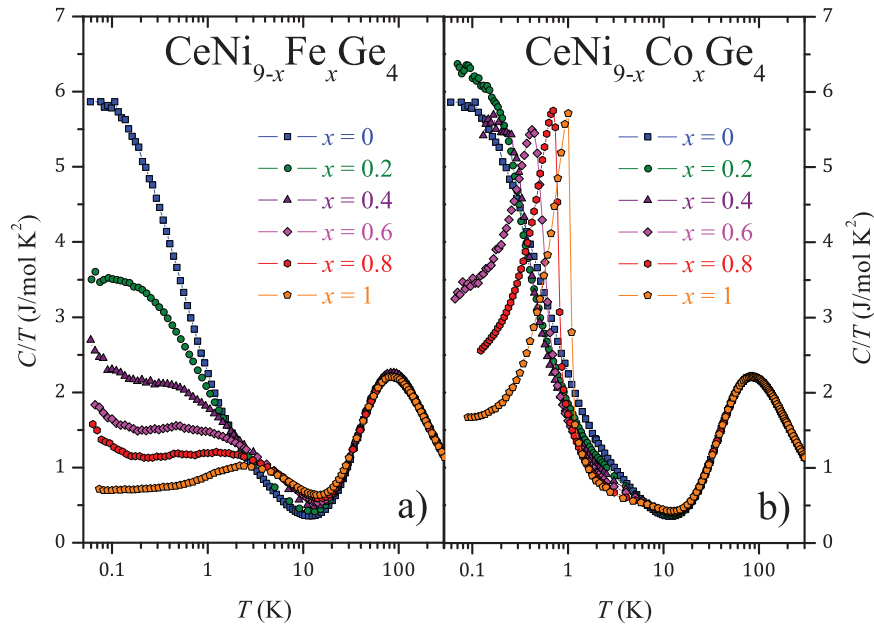


Fig. 3. The temperature dependence of the specific heat divided by temperature  $C(T)/T$  of (a)  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  and (b)  $\text{CeNi}_{9-x}\text{Co}_x\text{Ge}_4$  [13] in a semi-logarithmic plot.

$x$  signals a Kondo energy elevation. We further point out that the change of the  $T_{Rln4}$  values can be correlated with a transition from a  $SU(2)$  to  $SU(4)$  crossover regime for  $x=0$  to two well separated doublets ( $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$ ) for increasing concentration  $x$ .

In order to get more insight into the interplay between the competing energy scales, especially Kondo screening and CF splitting, in  $\text{CeNi}_{9-x}\text{T}_x\text{Ge}_4$  (T: Fe, Co, Cu) we introduce a macroscopic thermodynamic parameter  $\alpha$  which describes the resulting entropy temperature ratio between  $T_{Rln4}$  and  $T_{Rln2}$ . In the parent compound  $\text{CeNi}_9\text{Ge}_4$ , where Kondo screening and the subtle splitting of the two low energy Kramer's doublets coexist on the same energy scale, the ratio  $\alpha = T_{Rln4}/T_{Rln2}$  of 8.1 is accompanied by an effectively four-fold degenerate ground state. Upon Fe doping the  $\alpha$ -ratio first increases (even in the range of the error bars) and thus reflects disorder leading to an instantaneous increase

of the  $\Gamma_7^{(1)}$  and  $\Gamma_7^{(2)}$ -splitting observed in the case of the Co and Cu species. At larger Fe concentration ( $x > 0.2$ )  $\alpha$  drops rapidly down to 4.9 for  $x=1$ , whereas in the case of the Co and Cu series  $\alpha$  remains approximately constant at about 8.1 (Fig. 6). For the Fe samples this behavior might suggest that the Kondo temperature increases stronger than the  $\Gamma_7^{(1)}$ - $\Gamma_7^{(2)}$ -splitting energy and therefore the four-fold degenerate ground state appears to be retained over the concentration range up to  $x=1$ . For the Co substitution series, however, the Kondo temperature increases moderately in comparison to the CF splitting energy, resulting in a reduction of the effective spin degeneracy which finally leads to AF order. At least the AF order in the Cu series also suggests a reduction of the effective spin degeneracy. In this case a decreasing Kondo energy is attended by a strong  $\Gamma_7^{(1)}$ - $\Gamma_7^{(2)}$ -splitting energy [15].

Summarizing, the Fe substitution series  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  is one of the rare examples where a substitution driven increase of

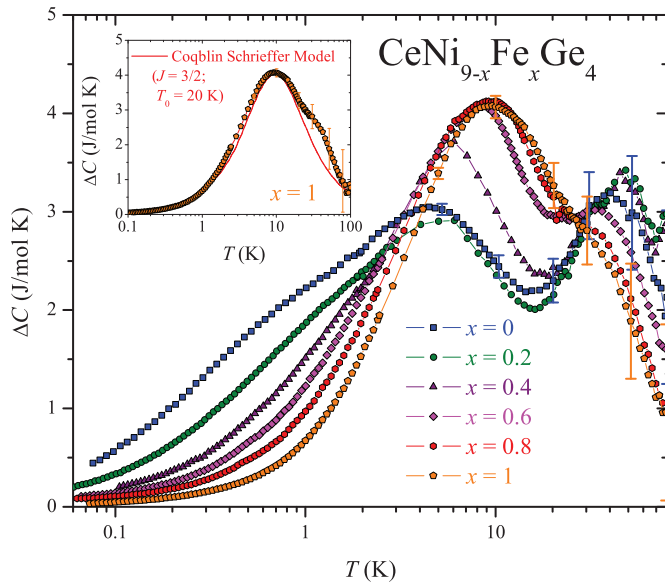


Fig. 4. The temperature dependence of the magnetic specific heat  $\Delta C$  of  $\text{CeNi}_{9-x}\text{Fe}_x\text{Ge}_4$  in a semi-logarithmic plot. Insert:  $\Delta C$  of  $\text{CeNi}_8\text{FeGe}_4$ . The solid line represents a Coqblin-Schrieffer model calculation [21] (see text).

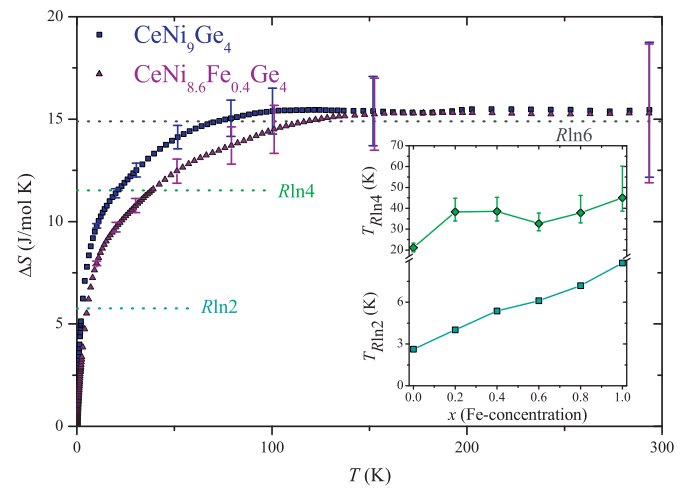
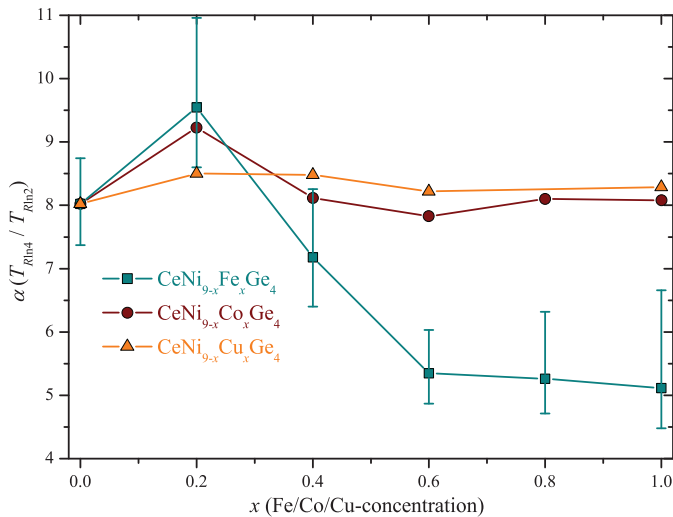


Fig. 5. The magnetic entropy  $\Delta S$  vs. temperature of the parent compound  $\text{CeNi}_9\text{Ge}_4$  in comparison to  $\text{CeNi}_{8.6}\text{Fe}_{0.4}\text{Ge}_4$ . The dotted lines mark the entropy limits for a doublet, quartet and sextet ground state degeneracy, respectively. Insert: Onset temperatures, where the entropy reaches the  $Rln2$  and  $Rln4$  limit with respect to the iron concentration,  $x$ .



**Fig. 6.** The entropy temperature ratio  $\alpha = T_{R\ln 4}/T_{R\ln 2}$  with respect to the Fe, Co [13] and Cu [12] amount  $x$ .

the ground state degeneracy is experimentally observed. The transformation of the ground state from the  $SU(2)$  to  $SU(4)$  crossover case in  $CeNi_9Ge_4$  to a well-defined  $N=4$  Kondo ground state for  $x \geq 0.4$  indicates that the increasing Kondo energy scale is the dominant electronic control parameter of the system, whereas, in the Co and Cu series the change of the ground state degeneracy is the main driving force for the development of AF order.

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