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Specific heat and magnetic susceptibility of CeNiSn doped with Rh

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Abstract
CeNiSn is known as a semimetallic system with a small pseudogap at the Fermi energy. We investigate the effect of Rh doping on the Kondo insulator CeNiSn by means of measurements of ac magnetic susceptibility and specific heat. We show that the formation of the Kondo insulator narrow gap in $CeNi_{1-x}Rh_xSn$ is associated with disorder-induced f-electron localization. For doped CeNiSn with $x \leqslant 0.06$, the electrical resistivity data follow an activation and variable range hopping behaviour at low T, consistent with weak disorder and localization, while C/T is large, which is not a common feature of Kondo insulators. For $x > 0.06$, the system is metallic and exhibits non-Fermi liquid behaviour with magnetic susceptibility $\chi \sim T^{-n}$ with $n \sim 0.4$ and electrical resistivity $\rho \sim T$.

1. Introduction

Kondo insulators are rare earth or actinide f-electron compounds with small-gap semiconducting properties [1, 2]. The gap of about 10 K originates from the hybridization between f-electrons and conduction electrons. Most Kondo insulators do not show magnetic order, exceptions being the ferromagnet UFe_4P_{12} [3] and the antiferromagnet SmTe [4]. It was reported [5] that the formation of the Kondo insulator (KI) gap (Δ) is due to the presence of a collective spin-singlet Kondo state, which is distinguished by a magnetic susceptibility $\chi(T) \rightarrow 0$ and activated behaviour of the electrical resistivity $\rho(T)$ with decreasing T. As a consequence, the universal scaling law $\rho(T)\chi(T) = \text{const}$ represents a universal characteristic of these strongly correlated electron systems and completes the definition of a Kondo semiconductor at $T \rightarrow 0$ from an experimental point of view [5]. The low-T electrical resistivity of CeNiSn approximately follows an exponential activation law [6], consistent with an energy gap in the density of states; however, this system is not a perfect semiconductor because there are impurity states in the bandgap and the gap is only a pseudogap [7]. We have shown that the Rh doping leads to the localization of f-electron states in CeNiSn and, as a consequence, a spin-glass-like state was observed in doped $CeNi_{1-x}Rh_xSn$ alloys for $x < 0.2$. We also showed that the gap is gradually closed by doping, yielding a metallic state for $x \geqslant 0.08$. Our systematic study has shown that the gap in doped CeNiSn is very sensitive either to the magnitude of the hybridization energy V or the change in carrier concentration, which is explained well within the framework of the periodic Anderson model [8].

In this paper, we present low temperature measurements of the specific heat for the Rh-doped Kondo semimetal CeNiSn. We found that the low-T specific heat coefficient, $\gamma \equiv C(T)/T$, is rather large and displays a temperature dependence characteristic of Landau–Fermi liquid (LFL) behaviour. The large value C/T is not a common feature of Kondo insulators and appears to be unique to Rh-doped CeNiSn.

2. Experimental details

Pure CeNiSn and CeRhSn samples were first prepared by arc-melting weighed amounts of the elemental constituents. Both samples were carefully examined by x-ray diffraction analysis and found to be single phase with the orthorhombic ϵ-TiNiSi structure (space group $Pnma$) and hexagonal structure of the Fe_2P type (space group $P\bar{6}2m$), respectively. The dilute $CeNi_{1-x}Rh_xSn$ alloys were then prepared by diluting nominal compositions of the master compounds. To ensure homogeneity, each sample was turned over and remelted several times and then annealed at 800 °C for 2 weeks. The samples were carefully examined by x-ray diffraction analysis and found to be single phase of the ϵ-TiNiSi type structure for

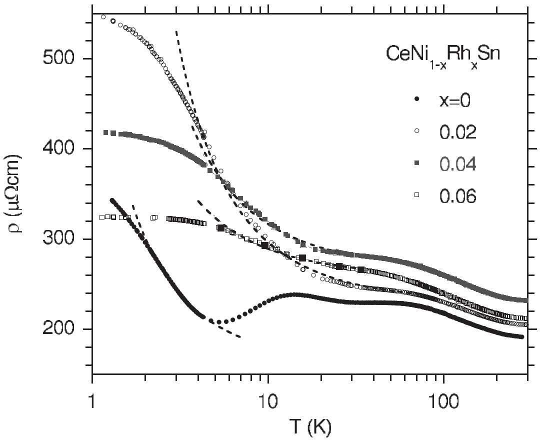

Figure 1. Temperature dependence of the electrical resistivity ρ for $CeNi_{1-x}Rh_xSn$ samples $x \leqslant 0.06$. The $\rho(T)$ curves exhibit an activated behaviour; however, the resistivity is also well fitted with a variable range hopping expression, $\rho = \rho_0 \exp(T_0/T)^{1/4}$ (dotted curve).

$x < 0.4$. The sample composition, determined using x-ray energy-dispersive spectroscopy, was found to be very close to the nominal value, consistent with negligible losses (less than 0.2% of the mass) that occurred during the melting process.

The specific heat was measured in the temperature range 370 mK–20 K and in external magnetic fields up to 8 T using a Quantum Design PPMS platform. We also used the PPMS for ac magnetic susceptibility measurements between 1.8 and 300 K.

3. Results and discussion

In figure 1, we display the temperature dependence of the electrical resistivity ρ for the samples with $x \leqslant 0.06$. The curves in figure 1 exhibit an activated behaviour; however, at low temperatures, the resistivity is also well fitted with variable range hoping: $\rho = \rho_0 \exp(T_0/T)^{1/4}$, with $T_0 \propto [N(E_F)\xi^3]^{-1}$, which indicates the existence of states with disorder in the gap. Here $N(F_F)$ is the density of states at the Fermi level and ξ is the localization length [9]. The fits are presented in the figure as dotted lines. The fits give a T_0 of about 5 K (table 1). The saturation of the resistivity below ~2 K may be due to states within the gap.

For the metallic regime (figure 2; $x > 0.06$), the resistivity displays a maximum at ~20 K and well-defined logarithmic dependence $\Delta\rho \sim -\ln T$ (not shown here), where $\Delta\rho$ is the difference between the resistivity of the sample and the resistivity of LaNiSn. The x-dependent maximum and the $\Delta\rho \sim -\ln T$ behaviour provide evidence that the maximum results from a competition between quantum coherence (i.e. itinerancy of f-electrons resulting from hybridization) and thermal disorder acting as a decoherence factor. Recently, we reported [7] $\Delta\rho \sim 1 + aT^n$ behaviour with $n \approx 1$ for $CeNi_{1-x}Rh_xSn$ at $T < 20$ K.

The low temperature specific heat data [10] show that CeNiSn is a metal with no long range magnetic order down to 25 mK with an enhanced density of states [*heavy-fermion (HF) type*] at the Fermi level. CeNiSn doped by Rh, on the other hand, has a large value of C/T which is strongly field-dependent. This is not a common feature of Kondo insulators or Kondo semimetals. In figure 3(*a*), $\Delta C/T$ exhibits pronounced maxima at ~7 K and ~0.7 K, respectively. The first feature is linked with pseudogap formation on the Fermi surface in $CeNi_{0.98}Rh_{0.02}Sn$. One should note that in the KI state the magnetic susceptibility of CeNiSn, after an impurity contribution has been subtracted, reaches a similar maximum [7] before passing into the $\chi \to 0$ limit with further decrease of T. The maximum in χ and $\Delta C/T$ at $T_m \approx$ 6 K determines the effective binding energy of a collective Kondo-singlet state in the Kondo insulating region (for details, see [5]). For the sample with $x = 0.02$, figure 3(*b*) displays the frequency-dependent maxima of the χ' and χ'' components of the ac magnetic susceptibility, characteristic of a spin-glass phase. These maxima at ~4.5 K appear, however, in a different

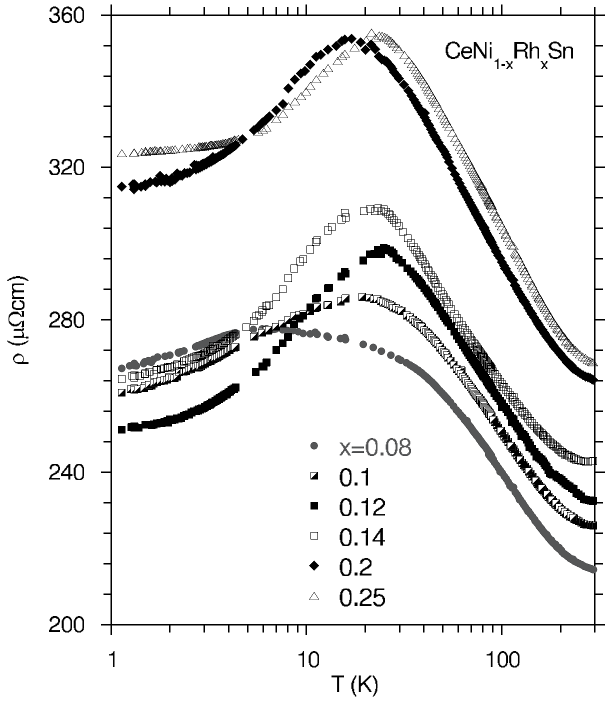

Figure 2. Temperature dependence of the electrical resistivity ρ for $CeNi_{1-x}Rh_xSn$, $0.08 \leqslant x \leqslant 0.25$.

Table 1. The electrical resistivity parametrization of the $CeNi_{1-x}Rh_xSn$ samples in the Kondo insulating region; the conductivity gap Δ is obtained from the best fit of the activated expression $\rho(T) = \rho_0 \exp(\Delta/T)$ to the experimental data. The activated behaviour is obtained in the temperature range 2–5 K for CeNiSn and between ~5 and ~20 K for the remaining $CeNi_{1-x}Rh_xSn$ samples. The electrical resistivity is also well fitted with variable range hopping, $\rho = \rho_0 \exp(T_0/T)^{1/4}$, in the temperature range 4 to ~30 K.

x	$\Delta(K)$	T_0 (K)
0	1.27	5.1
0.02	1.97	10.1
0.04	1.68	6.9
0.06	0.92	4.4

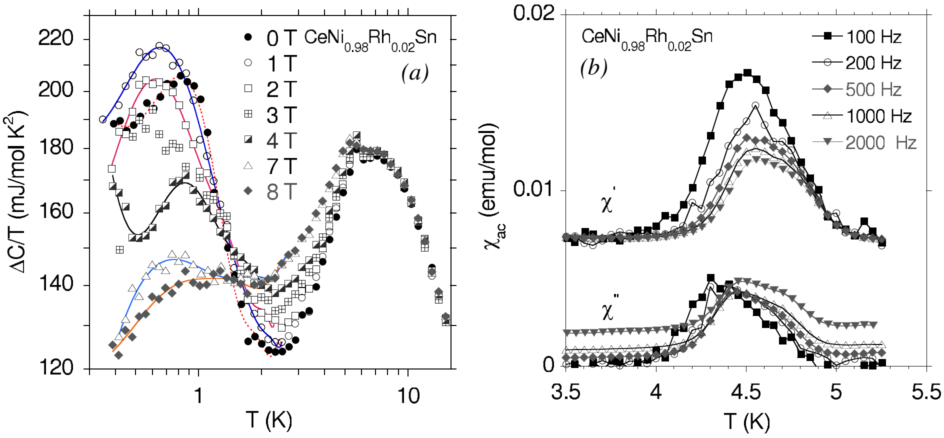

Figure 3. (*a*) Ce contribution to the specific heat of $CeNi_{0.98}Rh_{0.02}Sn$ as $\Delta C/T$ versus T on a log–log plot, for $B = 0$ and different magnetic fields. A kink at about 6 K is attributed to the extrinsic magnetic impurity phase Ce_2O_3 [11]. (*b*) Real χ' and imaginary χ'' components of ac magnetic susceptibility versus T measured at different frequencies ν in a field of 10 Oe.

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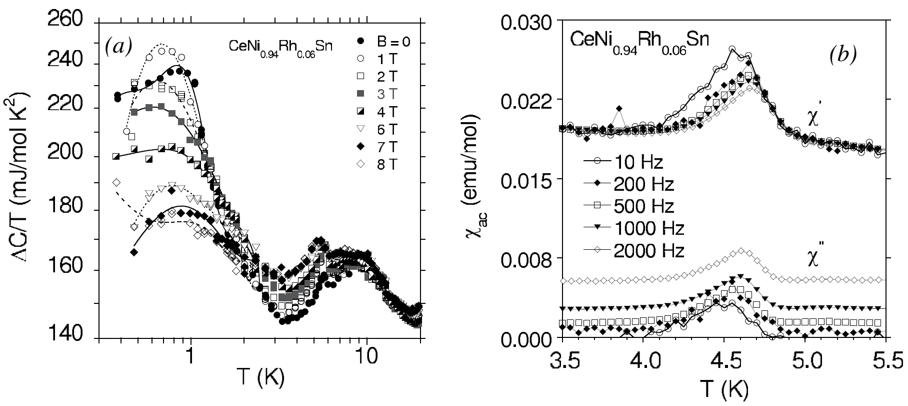

Figure 4. $\Delta C/T$ versus T on a log–log plot for $B = 0$ and different magnetic fields for $CeNi_{0.94}Rh_{0.06}Sn$ (*a*). Panel (*b*): real χ' and imaginary χ'' components of the ac magnetic susceptibility versus T, measured at different frequencies ν in a field of 10 Oe.

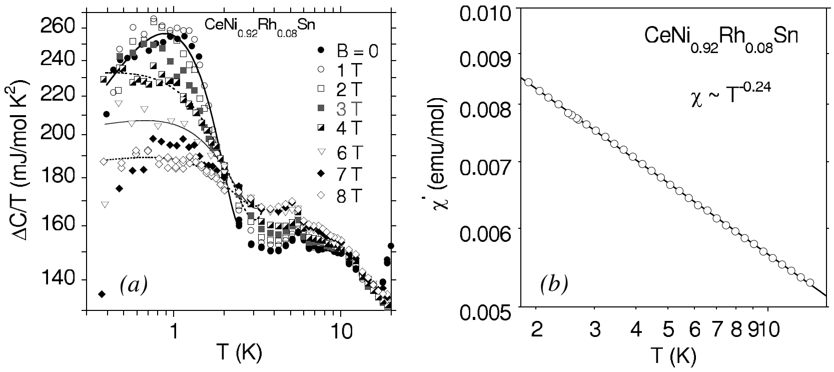

Figure 5. (*a*) $\Delta C/T$ versus T on a log–log plot for $B = 0$ and different magnetic fields for $CeNi_{0.92}Rh_{0.08}Sn$. The ac susceptibility does not show any frequency dependence (panel (*b*)); however, $\chi' \sim T^{-n}$ with $n = 0.24$. The concentration $x \equiv x_c = 0.08$ is critical (see text).

temperature range than the low-T rise in $\Delta C/T$ below 2 K. With this and the saturation of $\rho(T)$ at $T < \Delta/k_B$, the low-T rise in $\Delta C/T$ is indicative of Fermi liquid behaviour in the system with a small density of electrical carriers. The weak maximum at ∼0.7 K in $\Delta C/T$ instead of a saturation effect at the lowest temperatures can result from interplay of the FL with disordered magnetic moments. Similar dependences of both quantities $\Delta C/T$ and χ_{ac} are displayed in figure 4 for $CeNi_{0.94}Rh_{0.06}Sn$. Figure 4(*a*) clearly shows that the Kondo gap is strongly reduced for this compound.

Critical behaviour is seen for the sample with $x = 0.08$ in figure 5. In panel (*b*), the ac susceptibility does not show

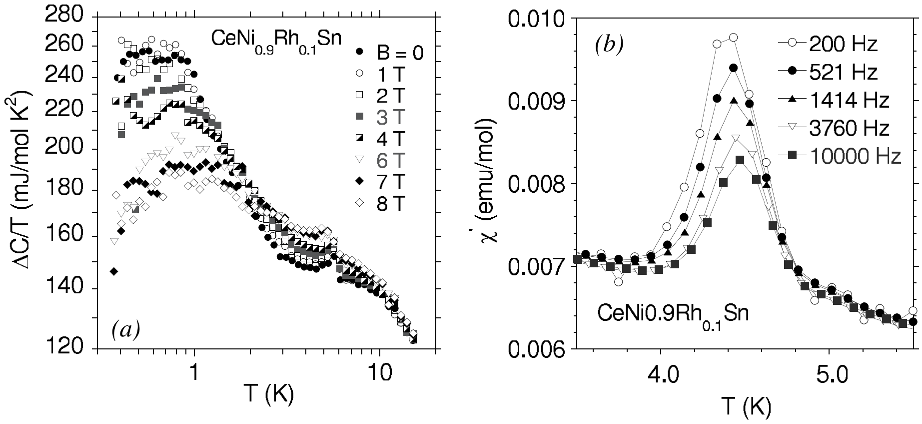

Figure 6. (a) $\Delta C/T$ versus T on a log–log plot at different magnetic fields for $CeNi_{0.9}Rh_{0.1}Sn$. Panel (b): ac susceptibility χ' versus frequency.

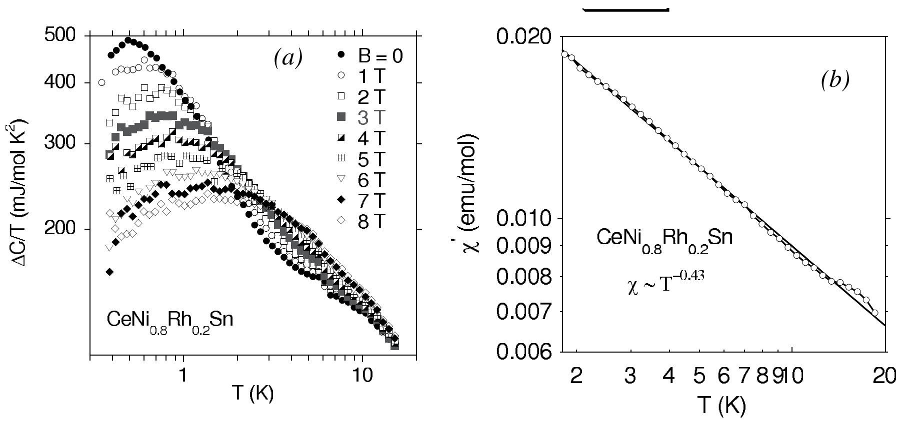

Figure 7. (a) $\Delta C/T$ versus T on a log–log plot at different magnetic fields for $CeNi_{0.8}Rh_{0.2}Sn$. The ac susceptibility (log–log plot) does not show any frequency dependence (panel (b)). $\chi' \sim T^{-n}$ with $n = 0.43$. The concentration $x \equiv x_c = 0.08$ is critical (see text).

any frequency dependence and $\chi' \sim T^{-n}$ with $n = 0.24$ in the temperature range 1.8 K $< T <$ 15 K. In panel (a), $\Delta C/T$ scales with the Kondo temperature T_K in the temperature range ∼1.2 K $< T <$ 3 K and can be described by the expression $\Delta C/T = -c \ln T/T_K$ in the limit $T < T_K$ with $T_K \approx 7$ K. Below 1 K, there is a crossover to a Fermi liquid phase. In the temperature region ∼7–15 K, the magnetic contribution to the specific heat (not shown in the figure) can be roughly compared to the theoretical ΔC curve of the $S = 1/2$ Kondo model [12]. Analysis of ΔC versus T data (not shown in the figures) confirms that $T_K \approx 7$ K, since in the broad ΔC Kondo peak $\Delta C_{max} \approx 1.5$ J mol^{-1} K^{-1} at $T_{max} = 0.45 T_K$ [13]. Thus, the Kondo effect appears to be responsible for this specific heat anomaly, when the gap is closed.

The maximum in $\Delta C/T$ at ∼6 K resulting from the formation of the Kondo semimetallic state is strongly reduced with increasing doping. The concentration $x \equiv x_c \approx 0.08$ seems to be critical, separating the KI and disordered metal regions. Figure 6 displays $\Delta C/T$ and χ' data for the sample with $x = 0.1$, which suggest the interplay of magnetic disorder and heavy Fermi-like behaviour. In figure 7, $\Delta C/T \sim -\ln(T/T_K)$ with $T_K = 6.2$ K for 0.5 K $< T <$ 3 K and χ' diverges. In an applied magnetic field, $\Delta C/T$ is suppressed and levels off towards a constant value as $T \to 0$, indicating recovery of Fermi liquid behaviour. On the other hand, the resistivity $\Delta\rho \sim 1 + aT^n$ and divergent character of χ observed for $x \geqslant 0.08$ can be attributed to quantum critical behaviour ($\chi = aT^{-n}$ with $n = 0.24$ and 0.43 for $x = 0.08$ or 0.2, respectively) in the entire metallic (NFL) regime $x_c \leqslant x < 0.4$.

In the Kondo semimetallic region, the entropy (figure 8) at $T_K \sim 7$ K is about $0.2R\ln 2$, which indicates that the low temperature specific heat data are associated with a Fermi liquid state. In the metallic state $S \sim \gamma T$, which is found for the system of $CeNi_{1-x}Rh_xSn$ alloys with low density of carriers. The inset displays the entropy of the samples with $x = 0.02$ and 0.2 in magnetic fields of 0 and 8 T. For both compounds, the magnetic field enhances the Fermi liquid state in the low temperature region. In a magnetic field of 8 T the Fermi liquid behaviour $S \sim \gamma T$ is more clearly visible. A slightly larger value of S of the sample with $x = 0.2$ compared to the entropy of the Kondo semimetals ($x \leqslant 0.06$) is consistent with the metallic character of this sample.

The large value of $\Delta C/T$ and the divergent character of the magnetic susceptibility indicate a different nature of the gap and the in-gap states of the Rh-doped CeNiSn than in normal Kondo insulators. Similar behaviour was recently

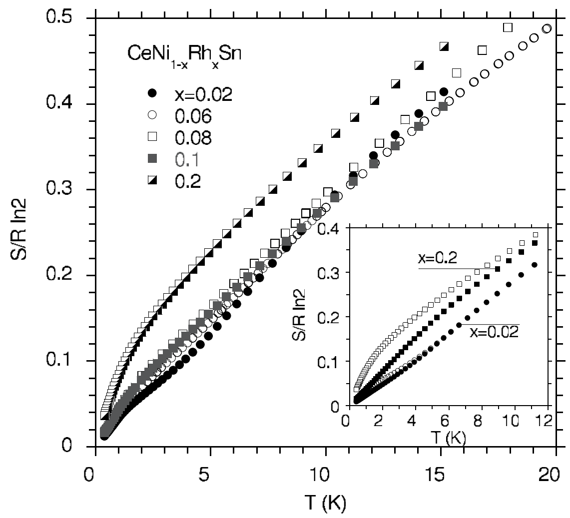

Figure 8. Magnetic entropy $S/R\ln 2$ for different $CeNi_{1-x}Rh_xSn$ samples versus temperature. The inset compares the entropy in zero magnetic field (open points) to the entropy measured in a magnetic field of 8 T (solid points) for samples with $x = 0.02$ and 0.2.

reported for $Ce_3Au_3Sb_4$ [14] when the f-band lies inside the gap. The density of in-gap states was suggested to be relevant to the hopping mechanism which then involves localized f-electrons. In the case of $CeNi_{1-x}Rh_xSn$ alloys, however, atomic disorder has crucial significance. As was mentioned, the resistivity of Rh-doped CeNiSn with $x < 0.06$ in the low-T region is well described by variable range hopping, which indicates the existence of localized f-states in the gap. It is quite likely that Ni/Rh atomic disorder at the 4c sites changes the J exchange coupling between Ce atoms, and this effect could result in the spin-glass behaviour in the $CeNi_{1-x}Rh_xSn$ series. In classical spin glasses, random freezing of the magnetic moments arises due to the dominant RKKY exchange interaction between the randomly situated magnetic moments. In the compounds studied, the Ce atoms occupy a periodic lattice, but the nonmagnetic elements (Ni, Rh, Sn), if they are disordered, introduce varying electronic environments around the Ce ions. As the Ce–Ce exchange interactions depend on the random occupation in the vicinity of the Ce ions, a spin-glass state would be possible at low temperatures, as shown in several other systems with structural disorder [15]. The atomic disorder strongly reduces the Kondo gap. For higher concentrations of Rh (i.e. for $x > x_c \approx 0.08$), the gap is completely suppressed and the system shows non-Fermi-liquid-like features. The second important effect leading to localization of the f-electron states is the change of hybridization energy V due to Rh doping. Our recent study [16] indicates a nonzero density of states (DOS) at the Fermi level for CeNiSn and a significant change of the hybridization energy V between the f-electron and conduction electrons when CeNiSn is doped with Rh. The stability of paramagnetic versus magnetic ground states in a Kondo insulator is strongly dependent on the magnitude of the on-site hybridization V and the number of valence electrons [8]. Thus the doping, even if small, increases the number of carriers and rapidly decreases the energy V. Therefore, a magnetic ground state could be formed in the $CeNi_{1-x}Rh_xSn$ series of alloys simultaneously with the appearance of the Kondo pseudogap.

4. Concluding remarks

CeNiSn is a Kondo semimetal with a low density of carriers at the Fermi level rather than a Kondo insulator. Thus, the low density of carriers plays a destructive role in the formation of the Kondo insulator ground state. We show that the Rh doping increases the density of carriers in $CeNi_{1-x}Rh_xSn$. On the other hand, the electrical resistivity, specific heat and magnetic susceptibility indicate that the formation of the inhomogeneous magnetic phase at $T <\sim 4$ K due to the effect of substitutional disorder, essentially for $x \geqslant 0.02$, is extremely important. Both effects remove the hybridization gap and give rise to a large heat capacity in the insulating state. This kind of behaviour (a very high heat capacity at low temperatures in the nonconducting state) is well known for the compounds with spin chains, which exhibit typical HF behaviour with large linear specific heat coefficient γ [17]. It should be noted that several physical quantities observed in these systems with charge (Yb_4As_3 [18]) or spin ($Y_{1-x}Sc_xMn_2$ [19]) degrees of freedom have shown similar heavy-fermion behaviour of extremely low carrier concentration. A giant γT term in the specific heat of these materials (very similar to that observed for $CeNi_{1-x}Rh_xSn$) is, however, not due to the usual Kondo-lattice mechanism, but has been discussed [20] as coexistence of an RVB type (resonating valence bound) state and magnetic frustrations. The study of Yb_4As_3 and P, Sb-doped mixed crystals [21] gave support to the existence of spin-glass behaviour in Yb_4As_3 at very low temperatures caused by the interchain coupling and disorder. The spin-glass effect gives a similar contribution to the low temperature specific heat of Yb_4As_3, as in the case of the doped CeNiSn. The *quasi-one-dimensional spin-chain systems* have, however, activation energy Δ about 100 times larger than the hybridization gap of the Kondo insulators. Thus, the possible magnetic frustration in CeNiSn caused by doping with Rh can easily destroy the coherent Kondo insulator gap and increase the number of the conduction electron states at the Fermi level. As a consequence, the upturn in the low temperature specific heat $C(T)/T$ data can result from both effects: short range magnetic ordering and Landau–Fermi liquid behaviour.

Recently, we argued [5] that, for Kondo insulators, the maximum of χ at T_m determines the effective binding energy of 4f spins into a singlet, while the energy $\Delta < T_m$ is connected with the binding energy of individual carriers into a collective singlet. For CeRhSb doped with Sn, we recently found [5] that T_m is evidently reduced when the doping is increased up to the critical concentration of ∼12%, which means that the destruction of the Kondo singlet with change of the number of valence electrons does not result from the change of the Kondo coupling, but it is rather due to change in the carrier concentration with doping. With increasing temperature, this collective singlet state is destroyed by the thermal motion at $T > T_m$. A similar effect can be observed when $x > x_c$. For $T_{coh} > T > T_m$, the f-electrons are itinerant, whereas for

$T < T_m$ the collective spin-singlet state causes a *reentrant localized* f-*electron behaviour*. In this scenario, the system would remain a heavy-fermion metal at $T \rightarrow 0$ if a collective spin-singlet state could not be formed due to an insufficient number of valence electrons. It could be a case of $CeNi_{1-x}Rh_xSn$, where replacement of Ni by Rh leads to a decrease of the number of valence electrons. At very low temperatures, the $C(T)/T$ data show Landau–Fermi liquid behaviour due to a residual density of carriers at ϵ_F. At the lowest temperatures, $C(T)/T$, however, does not saturate. We interpret this anomaly in the specific heat as a result of the interplay of LFL and the weak spin-glass intrinsic effect with the maximum in χ_{ac} at ~4.5 K. The in-gap localized f-electron states are formed at low temperatures as a result of disorder. In the disordered $CeNi_{1-x}Rh_xSn$, there are two possible mechanisms which lead to localization of the f-electrons: variable range hopping and/or the nonmagnetic atomic disorder, NMAD, mechanism, or both. In effect, weak inhomogeneous magnetism appears, which can remove the gap in $CeNi_{1-x}Rh_xSn$.

The effect of localization of the f-electron states in a Kondo insulator can be understood in the Anderson model. It was shown that stability of the paramagnetic versus magnetic ground state in the Kondo-lattice limit [8] is strongly dependent on the magnitude of the on-site hybridization V and the number of electrons n_e. Doradziński and Spałek [8] (DS) discussed the possible magnetic phases in the periodic Anderson model and obtained a phase diagram in the V–n_e plane which provides a qualitative account of experimental results on a series of Ce-ternary intermetallics [22]. In the DS diagram, CeNiSn is located on the line $n_e \approx 2$ in the Kondo insulator region, very close to the critical value of $V \approx 0.3$ eV, which separates the KI and antiferromagnetic Kondo insulator (AKI) phases. Our recent XPS measurements revealed that the hybridization energy V is strongly reduced in CeNiSn with Rh doping [16] when the concentration $x > 0.06$. This experiment implies that doping can easily vary the KI state to the state with the localized f-electrons and simultaneously removes the Kondo gap. On the basis of the simple theoretical model of DS, one can qualitatively explain the existence of the short range ordering or spin-glass state in the Kondo insulator limit, when the number of electrons n_e is almost two.

The interesting question is whether the critical concentration x_c is associated with the quantum critical point. In the phase diagram (see [7]), this concentration separates the KI phase, in which there is an interplay with the weak spin-glass type behaviour, from the metallic phase with a weak magnetic inhomogeneity. At this point, the susceptibility does not show any frequency dependence and $\chi \sim T^{-n}$, $\Delta\rho \sim T$, while $\Delta C(T)/T \sim \ln(T/T_0)$. We also note that these quantities display different behaviours on the right and left side of x_c. It seems to be possible that $x_c \approx 0.08$ is a quantum critical point: However, the explanation of the nature of this unique behaviour will require further investigations.

Acknowledgments

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