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Mössbauer investigations of the FeCoNiPd_x and $\text{Cr}_x\text{FeCoNiPd}$ high entropy alloys

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Abstract

Two series of FeCoNiPd_x and $\text{Cr}_x\text{FeCoNiPd}$ high entropy alloys were investigated using SEM-EDX, XRD technique and Mössbauer spectroscopy. Increasing of Pd content in the FeCoNiPd_x samples only slightly influenced the Mössbauer spectra. They were analysed assuming simultaneous presence of hyperfine field and quadrupole splitting distributions, however random direction of observation relative to H and EFG caused the average QS value to be close to zero. Increasing of Cr content in the $\text{Cr}_x\text{FeCoNiPd}$ samples resulted in strong decrease of the hyperfine field, its distribution becomes wide and influence of H , $IS(H)$ and EFG on the Mössbauer spectra can not be separated unambiguously. The decomposing of the HEAs compositions into binary artificial Fe-dopant systems, namely $[\text{FeCoNi}]\text{-Pd}$ and $[\text{FeCoNiPd}]\text{-Cr}$, allowed to interpret the magnetic structure of the two investigated series.

1. Introduction

Physical properties of iron based binary systems have already been studied in various concentration ranges. Mössbauer technique is particularly useful as it allows very local determination of the impact of the dopant on the properties of Fe or also Sn nuclei in the crystal structure ([1] and references therein). It is well known that various elements can strengthen or weaken magnetic properties of an alloy, depending on the concentration of dopants, their distance to the ^{57}Fe -atom and their arrangement. Depending on the ratio of the constituents in the alloy, different models and techniques of Mössbauer spectra analysis are used ([2, 3] and references therein).

High entropy alloys (HEAs) differ from classical alloys in the fact that all constituents are major [4, 5, 6]. Thus, no constituent can properly be called dopant. However, to extend the issue of additivity of interactions and potential transfer of relationships determined in binary systems to more complex ones could add to the understanding of HEAs because these alloys are characterized by the simplicity of their crystal structure. On the other hand, strong disorder characterizing HEAs and multiplicity of concurrent admixtures allow to study the effect of various atom configurations around iron on its hyperfine parameters. HEAs containing palladium are very attractive in this respect, because unlike most of the systems studied, they do not change their crystal structure in a very large Pd concentration range, and more importantly, no precipitation of new phases with

clearly different stoichiometry is observed [7, 8, 9, 10]. This gives the unique opportunity to track the impact of the admixture on the alloy's physical properties over a wide concentration range. In this work, two systems were selected for detailed investigations, with varying Cr or Pd contents. In binary alloys Cr and Pd presence in the vicinity of iron, change Fe hyperfine parameters in opposite ways, namely decreasing or increasing their values. In this study in order to apply the binary systems approach the compositions of the investigated alloys have been artificially decomposed into two parts: i) the major constituent of the HEA acting as "dopant", namely Cr or Pd and ii) the rest of the constituent elements, including Fe, playing the role of Fe in binary systems.

2. Samples preparation and experimental methods

Two series of samples were prepared: i) one with no Cr and variable Pd content, S_{Pd} : FeCoNiPd_x , $x=0.5, 1.0, 1.5, 2.0$, and ii) one with constant Pd and variable content of Cr, S_{Cr} : $\text{Cr}_x\text{FeCoNiPd}$, $x=0.0, 0.5, 1.5, 2.0$. Thus, the FeCoNiPd_1 alloy is common to both series. The samples were prepared by melting pure elements in an arc furnace under the Ar atmosphere. They were remelted four times, flipping ingots between remelts to ensure their homogeneity. A thin metal flake ($\sim 0.3\text{mm}$ thick) was cut from each ingot and further rolled to a thickness of $30\text{-}35\mu\text{m}$. 10mm diameter circles were cut from the obtained foils and homogenized in vacuum at 1000°C for 24h. The annealing was completed by quenching the samples onto a solid piece of brass. Such prepared samples were used to perform SEM-EDX, XRD and Mössbauer measurements.

The samples XRD patterns were collected at RT with a PANalytical Empyrean diffractometer using a diffracted beam graphite monochromator and an X'Celerator linear detector (Cu K α radiation) scanning the 2Θ -range from 10° to 150° in steps of 0.033° . Diffraction patterns were analyzed by the Rietveld method as implemented in the FULLPROF program [11].

The microstructure observations and chemical analyses were carried out in a FEI Versa 3D scanning electron microscope equipped with an EDAX Apollo XP energy dispersive X-ray spectrometer (EDX), operated at 20 kV accelerating voltage.

Mössbauer spectra were collected in transmission geometry using standard spectrometer in the constant acceleration mode. A mobile source of $^{57}\text{Co}(\text{Rh})$ γ -ray photons was kept at room temperature.

3. Results and discussion

Using the SEM-EDX technique, average compositions were determined for each sample. In four samples: $\text{Cr}_x\text{FeCoNiPd}$, $x=0.5, 1.5, 2.0$ and $\text{FeCoNiPd}_{0.5}$, slight fluctuations in the composition of some elements were observed. In all cases the concentration of Fe and Cr remained constant, fluctuations were observed for palladium but also nickel and cobalt. In all cases, the oscillation length was similar, $\sim 50\mu\text{m}$, and the amplitudes amounted to $\sim 2\text{-}3\text{at}\%$ for Pd and to $\sim 1\text{-}1.5\text{at}\%$ for Co and Ni. The observed composition fluctuations are small and were not detected using XRD technique. It is known that the alloys of FeCrCoNiPd-HEA system crystallize in one *fcc* structure. However, with the use of high energy XRD technique several *fcc*-phases with slightly different lattice parameters were detected [12, 7, 8, 9, 10]. The detailed investigation of the structure and microstructure of the alloys of this study are presented in forthcoming papers [13].

Based on the analysis of the diffraction patterns it was found that all correspond to a single *fcc* structure, independent on the composition. It was assumed in the analysis that each crystallographic site was occupied by all constituent elements with probabilities corresponding to the compositions

as determined by SEM-EDX measurements. In the samples of the S_{Pd} series, a clear increase of the average lattice parameter versus palladium content is observed (Fig. 1), which is expected because of the significantly larger palladium atom size (137.6 pm) as compared to the one of the other elements (124.7-128.2 pm)[14]. This has earlier been observed in [8, 9]. In samples of the S_{Cr} series the lattice constant slightly decreases with the increased Cr content (Fig. 1).

Changes in iron hyperfine parameters caused by a small (<10at%) admixture of another element are usually analyzed with the following additive model:

$$X(n_1, n_2, n_3, \dots) = X_0 + \Delta X_1 \cdot n_1 + \Delta X_2 \cdot n_2 + \Delta X_3 \cdot n_3 + \dots$$

where X stands for the quantity analyzed (hyperfine field H , isomer shift IS or quadrupole splitting QS), n_i are quantities and ΔX_i are corrections due to the non-ferrous atoms located in the i -th coordination sphere. However, the applicability of this model for higher dopant concentrations is disputable, and it certainly breaks down after the sample goes into a paramagnetic state. Thus, in the present investigations, the artificial binary systems are [FeCoNi] - Pd and [FeCoNiPd] - Cr where [FeCoNi] and [FeCoNiPd] systems take the role of Fe while Pd and Cr the role of the dopant, in [Fe-dopant] binary systems. The elements of the HEAs analyzed here increase (with exception of Cr) the hyperfine field on iron atoms. The corresponding ΔH_i -values at room temperature are summarized in the Table 1. Since there is no chromium in the samples of S_{Pd} series the alloys are expected to stay magnetically ordered at room temperature in accordance with conclusions from the analysis of binary alloys. In the samples of the S_{Cr} series the chromium content is continuously increasing. The expected result is a decrease of the average hyperfine field on Fe.

The measured Mössbauer spectra of the S_{Pd} series are shown in Fig. 2. In all cases, a magnetic splitting and a distribution of the hyperfine field, hfd, can be observed. However, the sextuplet lines are clearly separated. The spectra are all similar and in all cases the same asymmetries in both amplitude and width of the corresponding line groups are seen: the first line group is clearly wider and shallower than the sixth one. The second line group is deeper and narrower than the fifth one. Similarly, the third one is deeper and narrower than the fourth one. The asymmetries observed for line groups 1 and 6 are opposite to those observed for the other groups which indicates that they are caused by quadrupole splitting which affects them in the opposite way. On the other hand, there is no visible shift in the average position of the line groups on the velocity scale that would result from the existence of a fixed QS value.

A very similar character of the Mössbauer spectra was noticed for FePd alloys [15, 16, 17, 18, 19, 20], with high Pd contents. In this system, the spectra differed in the values of the hyperfine parameters, and for some concentrations magnetic splitting was observed only at low temperatures. However, it should be emphasized that in all cases the same asymmetry features of the line groups were observed. The spectra were analyzed and explained based on the presence of the electric field gradient, EFG , and consequently the presence of QS .

In this paper all Mössbauer spectra of the S_{Pd} samples were analysed assuming a single IS -value for the whole spectrum (i.e. without isomer shift distribution, or $IS(H)$ -dependence). Clebsch-Gordan coefficients were determined to 1 : 2.26 : 3. For a purely random orientation the 1 : 2 : 3 relation would be observed. This deviation indicates a slight preference for magnetic field orientation, possibly the consequence of the texture residue resulting from the cold rolling of the samples. For the cold rolled but not annealed sample, not shown here, the Clebsch-Gordan coefficients are found to be 1 : 3.6 : 3. Based on the qualitative features discussed above, a spectrum analysis model was proposed, consisting of the coexistence of the hyperfine field distribution and the quadrupole splitting distribution, with the mean value of the latter close to zero. All four spectra were analysed

with this model. The two-dimensional H and QS distributions are presented in Fig. 2 and the corresponding average parameters in Fig. 3. It can be observed that the average value of the hyperfine field changes only slightly with the increase in Pd content and that all H distributions are relatively narrow. The QS distributions are symmetrical and although the average values of $|QS|$ are very small they are measurable (see Fig. 3).

In the case of the S_{Pd} series, surprisingly high values of the hyperfine fields were detected, despite of the fact that iron constitutes only $\sim 20 - 30\%$ of the alloy. This can be attributed to the elements whose presence in the crystal structure in the vicinity of the iron atom increases its hyperfine field. Moreover, as a result of the proximity to the iron atom in the first or second coordination shell, all three constituents increase the H by a similar value ($\sim 8-12$ kOe), which induce the narrowing of hyperfine field distributions. Besides, the effect of the relative decrease of iron content and the increase of non-iron neighbors is superimposed by an increase of the atomic distances (caused by the presence of palladium in the lattice), which stems an increase of the magnetic properties. As a combined result, only a slight decrease of the average field is observed.

Because the palladium atoms are clearly larger than the other constituent elements, their increasing amount causes an increase in the disturbances of the local atomic positions, even if on the average the structure of fcc is still present. The disturbed local cubic symmetries around the ^{57}Fe atom cause the appearance of a non-zero EFG . As a consequence a quadrupole splitting co-existing with the magnetic field is observed. Since palladium atoms in the lattice are a source of both quadrupole splitting and hyperfine field disturbances, correlations between these quantities are expected. However, the orientation of the EFG and H in relation to the direction of the gamma rays (observation direction) during the measurement is random. Therefore, the average QS value in the observed Mössbauer spectrum is close to zero.

The Mössbauer spectra of the S_{Cr} series are presented in Fig. 4. A very pronounced decrease of the average hyperfine field is observed with increasing Cr content and a relative decreasing effect of palladium, which is the result of the dilution mentioned above. In a binary alloy, the presence of chromium in the nearest (NN) or next-nearest (NNN) neighbor's position causes a decrease of the hyperfine field on iron by ~ 31 and ~ 21 kOe respectively, i.e. 2-3 times more than the increase caused by the proximity of other admixtures [21]. In the present study these factors cause the hfd to widen and the rapid (with an increase in Cr content) drop in \bar{H} , so that the last sample of this series with the highest chromium content, $\text{Cr}_2\text{FeCoNiPd}$ is paramagnetic at room temperature. The spectra of the S_{Cr} series samples can be analyzed with a model similar to the one proposed for the S_{Pd} series, that is $[\text{FeCoNiPd}]\text{-Cr}$. The results are the following: 1) The decreasing values of the hyperfine fields and the expansion of the distribution caused by the increasing presence of chromium allow to analyze the asymmetric spectra based on the standard hyperfine field distribution model. Cr atoms in the vicinity of Fe change both the charge and spin density ([22] and references therein). Consequently, an $IS(H)$ relationship appears in the Mössbauer spectrum. 2) Due to the removal of QS degeneration caused by H disappearance, the spectrum of the $\text{Cr}_2\text{FeCoNiPd}$ sample is a doublet (the quadrupole splitting distribution curve is presented in the top right of Fig. 4). This suggests QS presence in all samples of this series. 3) The structure of the spectra is not clear due to the broadening of the distribution and the decrease of the H value. This results in the overlap of the first and second lines in the spectrum and their unbalanced asymmetry. As a consequence, it is not possible to clearly separate the impact of all three hyperfine parameters based on their shape. 4) The Mössbauer spectra of the samples of this series for which magnetic splitting is visible were analysed in terms of double quadrupole splitting and hyperfine field distributions (see Fig. 4). It should be noted here, that other fitting models (e.g. with $IS(H)$ -dependence) would also

be effective, but there is no any indications which of them would be correct. On the other hand, obtained \overline{H} and \overline{IS} -values are generally model independent.

Although Mössbauer spectra of both series do not have a clear structure and can only be analyzed using hfd, comparing the average distribution parameters with the values obtained for binary systems allows conclusions to be drawn regarding the influence of neighbors on the hyperfine field observed on iron. For this aim, the artificial binary systems defined above were used. The relationships are compared in Fig. 5 for both series. Fig. 5a presents the series with Pd as "dopant" as compared with real binary systems from the literature [15, 16, 17, 19, 23]. Fig. 5b presents the second series with Cr as "dopant" as compared with binary real Fe-Cr alloys measured with the same installation [24]. It can be observed that the \overline{H} -value changes are in both series very similar to those corresponding to the real binary systems, namely Fe-Pd and Fe-Cr, when compared versus the concentration of "dopant". When the decomposition into artificial systems is made based on Fe, not on the dopant, namely, Fe-[CoCrNiPd] and Fe-[CoCrNi], there is no agreement between the obtained data. Accordingly, the artificial HEA binary systems for which the relationships can be interpreted as a binary Fe-dopant system are the following: $(\text{FeCoNi})_{1-x}\text{Pd}_x$ vs $\text{Fe}_{1-x}\text{Pd}_x$ and $(\text{FeCoNiPd})_{1-x}\text{Cr}_x$ vs $\text{Fe}_{1-x}\text{Cr}_x$.

It can be concluded that the effect of cobalt and nickel on the hyperfine field on Fe atoms is small in these HEA systems and that these elements are "seen" as iron atoms in NN or NNN positions. Pronounced changes occur only when chromium or palladium appears in the vicinity of the Fe atom. Moreover, these changes are very similar to those observed in binary systems. This may be due to the relatively small impact of Co and Ni admixtures in the analyzed concentration range. This is shown in the Table 1, where the values of ΔH_{Co} and ΔH_{Ni} are relatively small. Their interaction may also have a cancelling character (in all cases Co and Ni admixtures occur in the same 1 : 1 ratio). Detailed explanation of the effect of individual atoms neighborhood will be analyzed and presented in forthcoming papers.

4. Conclusions

Based on the measurements performed using EDX, XRD and Mössbauer spectroscopy on two series of FeCoNiPd_x and $\text{Cr}_x\text{FeCoNiPd}$ high entropy alloys, the following conclusions can be drawn:

- The decomposing of the HEAs compositions into binary artificial Fe-dopant systems, namely [FeCoNi]-Pd and [FeCoNiPd]-Cr, allowed to interpret the magnetic structure of the two investigated series. This also indicates that the influence of cobalt and nickel on the hyperfine field on Fe atoms is small in these systems.
- Increasing of Pd content in the FeCoNiPd_x samples results in the clear increase of the lattice parameter, but only slightly influences the Mössbauer spectra. All the samples are magnetic at room temperature and present simultaneous hyperfine field and quadrupole splitting distributions. The effect of the increasing amount of dopants on the hyperfine field on Fe atoms is compensated by the increase in atomic distances. The random direction of observation relative to H and EFG causes the average QS value to be close to zero.
- Increasing of Cr content in the $\text{Cr}_x\text{FeCoNiPd}$ samples results in the small decrease of the lattice parameter, and strongly influences the Mössbauer spectra. Presence of Cr atoms in the first or second co-ordination shell of Fe atom decreases its hyperfine field by different amounts. Hyperfine field distribution becomes wide and influence of H , $IS(H)$ and EFG on the Mössbauer spectra can not be separated unambiguously.

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Table 1: Corrections of the hyperfine field by single dopant atom located in the first (ΔH_1) and second (ΔH_2) co-ordination sphere (in kOe).

element	ΔH_1	ΔH_2	ref.
Cr	-31.0	-21.3	[21]
Co	9.0	9.09	[25]
Ni	7.25-7.55	—	[26]
Pd	10.8-12.8	5.1-9.8	[27, 23]

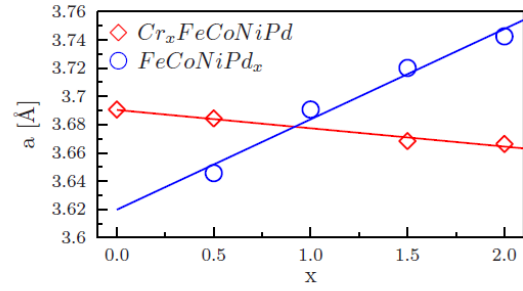


Figure 1: Average lattice parameters determined from the XRD patterns for the FeCoNiPd_x and Cr_xFeCoNiPd-HEA alloys.

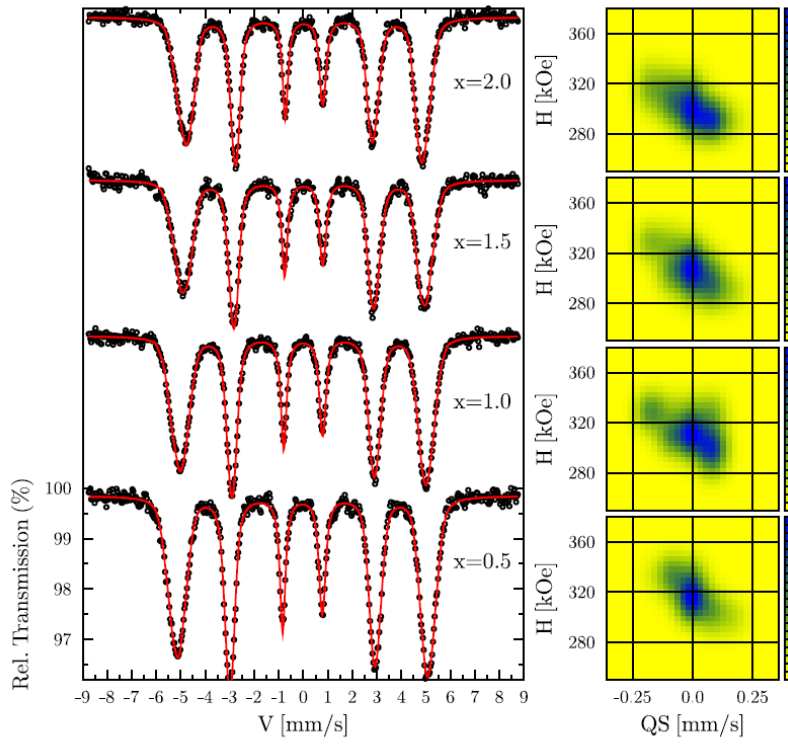


Figure 2: Room temperature Mössbauer spectra for $\text{FeCoNiPd}_x\text{-HEA}$ samples (left) and corresponding hyperfine field - quadrupole splitting distribution plots (right).

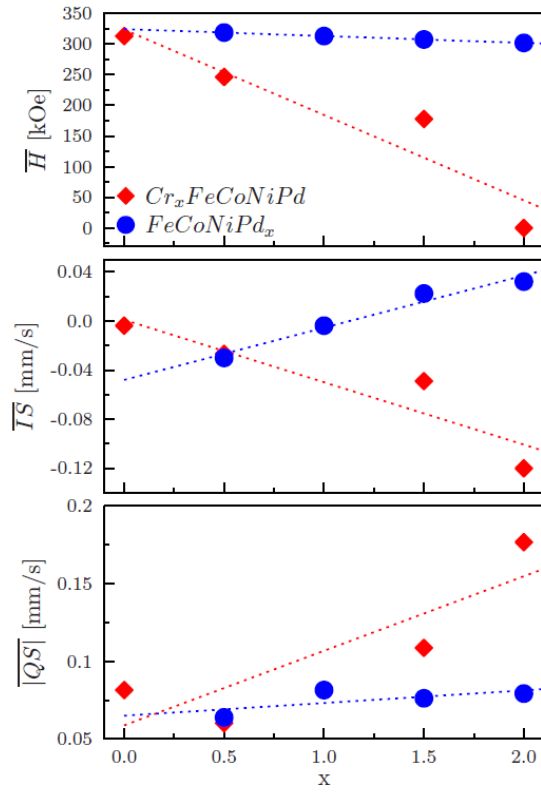


Figure 3: Hyperfine parameters of the $FeCoNiPd_x$ and $Cr_xFeCoNiPd$ -HEA alloys: average hyperfine field \overline{H} , isomer shift $\overline{I_S}$ and absolute value of quadrupole splitting, $|\overline{Q_S}|$.

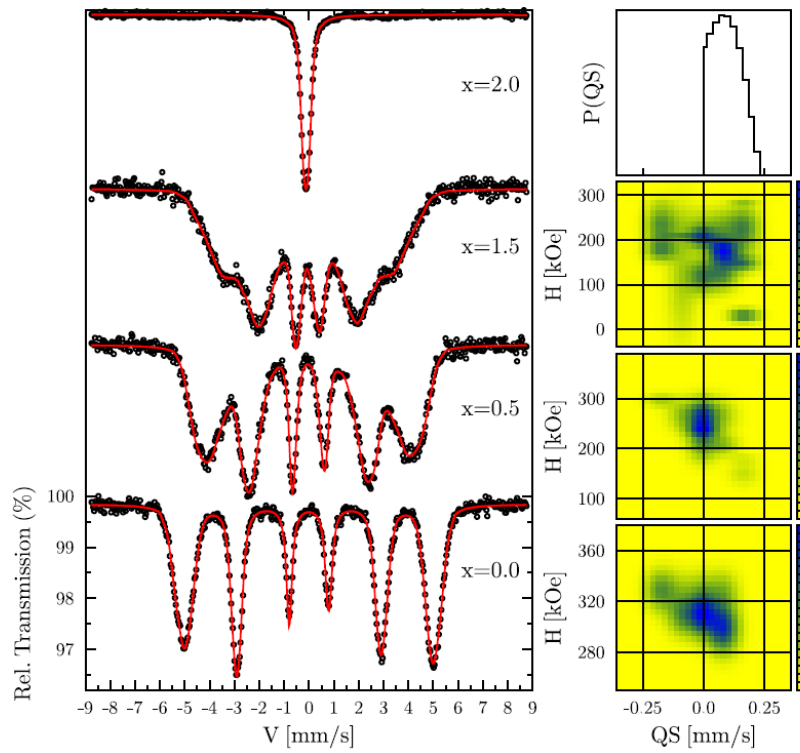


Figure 4: Room temperature Mössbauer spectra for $\text{Cr}_x\text{FeCoNiPd}$ -HEA samples (left) and corresponding hyperfine field - quadrupole splitting distribution plots (right). At the top right, quadrupole splitting distribution curve corresponding to $\text{Cr}_2\text{FeCoNiPd}$ sample is shown.

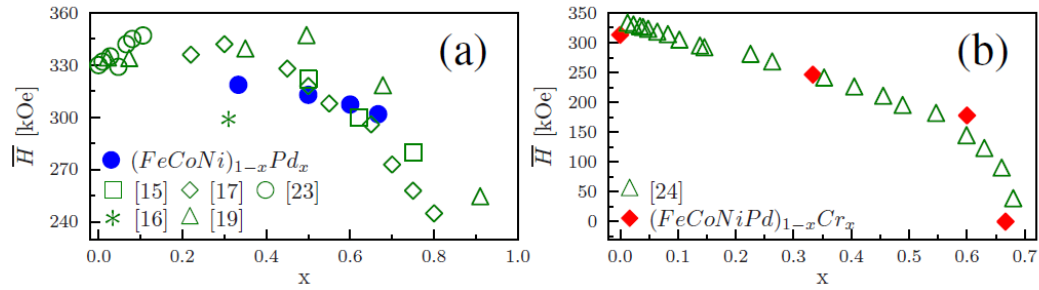


Figure 5: Average hyperfine fields of two analysed HEA-series as compared to similar Fe binary systems: (a) $(\text{FeCoNi})_{1-x}\text{Pd}_x$ and $\text{Fe}_{1-x}\text{Pd}_x$ and (b) $(\text{FeCoNiPd})_{1-x}\text{Cr}_x$ and $\text{Fe}_{1-x}\text{Cr}_x$. Full symbols stand for the values obtained for HEA systems in this work, empty symbols denote literature data.

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