



*‘I am among those who think that science has great beauty.  
A scientist in his laboratory is not only a technician:  
he is also a child placed before natural phenomena which  
impress him like a fairy tale.’ Marie Curie*

# A “multielement cocktail”

## Magnetism in nanocrystalline high-entropy alloys

By Natalia Shkodich

The traditional concept of alloy design that has been in use from the Bronze age up to the present day is based on the selection of one (or rarely two) principal element (e.g. Fe-based steels or Ni-based superalloys), while other elements are usually added in low concentrations for properties enhancement (Fig. 1a). The high concentration of a principal element (usually more than 50 at%) determines the overall properties of the material as well as the crystal lattice, and thus the regular internal arrangement of the atoms in the material. In steels, for

example, iron as the main element usually has a mass fraction of more than 60–70 %. An element such as chromium is commonly added to enhance the corrosion resistance of alloys, tungsten is added to improve wear resistance at high temperatures, and carbon is frequently added to improve strength. The regions of the corners (next to the single element) and edges (binary alloys) of the multicomponent phase diagram (Fig. 1b, insert) have been studied extensively in the past.

A fundamentally novel strategy for exploring unknown regions in

multicomponent phase diagrams (Fig. 1b, insert), where alloys consist of at least five principal elements in near equiatomic amounts, has been discussed and explored since 1995. In 2004, this was introduced independently by Prof. Yeh et al. [1] and Prof. Cantor et al. [2] as high-entropy alloys (HEAs).

The presence of multiple principal elements (five or more) in HEAs is not just a difference in composition, but also has fundamental physical effects on the configurational entropy, phase selection/stability and Gibbs free energy.

Let us briefly consider what the main idea is based on. From thermodynamics, it is known that a system tries to minimize its Gibbs free energy ( $\Delta G_{\text{mix}}$ ) to achieve a stable or metastable equilibrium state. Enthalpy of mixing ( $\Delta H_{\text{mix}}$ ) and total mixing entropy ( $\Delta S_{\text{mix}}$ ) are related to Gibbs free energy at a given temperature (T), shown by the equation:

$$\Delta G_{\text{mix}} = \Delta H_{\text{mix}} - T\Delta S_{\text{mix}} \quad (1)$$

Decreasing the  $\Delta H_{\text{mix}}$  or increasing the  $\Delta S_{\text{mix}}$  can decrease the  $\Delta G_{\text{mix}}$ . The mixing entropy  $\Delta S_{\text{mix}}$  has four major contributions: configurational, vibrational, magnetic dipole and electronic randomness. Among high-entropy alloys, the configurational entropy is much higher than with conventional alloys (which typically have one or two principal elements). The phase selection is determined by the competition between  $\Delta H_{\text{mix}}$  and  $T\Delta S_{\text{mix}}$  (unless the strain energy effect resulting from the atomic size difference is considered).

When chemical disorder is introduced by mixing multi-principal elements, the  $\Delta S_{\text{mix}}$  increases, especially at high temperatures, and thus the  $\Delta G_{\text{mix}}$  is reduced (equation (1)). That means that the phase separation of chemically-ordered intermetallic compounds energetically become less favoured. As a consequence, simple solid solutions with homogeneous face-centered cubic (*fcc*), body-centered cubic (*bcc*) and hexagonal closest packed (*hcp*) structures are formed.

The specific features of the chemical composition and structure lead to four ‘core effects’ observed in HEAs, which determine the unique properties of these materials: *high-entropy*, *lattice distortion*, *sluggish diffusion* and *cocktail effects*.

Let us briefly consider each of them:

The *entropy effect* achieved by mixing multiprincipal elements (five or more) can stabilize the disordered solid solutions in alloys and prevent the formation of chemically ordered intermetallic compounds. The formation of solid solution phases in HEAs

is governed by the *entropy of mixing*  $\Delta S_{\text{mix}}$ , and *enthalpy of mixing*  $\Delta H_{\text{mix}}$  according to equation (1).

The mixing entropy increases as the number of elements increases according to the equation:

$$\Delta S_{\text{mix}} = -R\sum_{i=1}^n C_i \ln C_i \quad (2)$$

Here,  $R$  is the gas constant of 8.314 J/(Kmol) and  $C_i$  is the molar fraction of the  $i^{\text{th}}$  element.

Based on the value of  $\Delta S_{\text{mix}}$  (in other words, on the number (N) of principal elements in the alloy), the literature contains the following classification of alloys (Fig. 1b): alloys with  $\Delta S_{\text{mix}} \leq 0.69R$  are referred to as low entropy (traditional), alloys with  $0.69R < \Delta S_{\text{mix}} < 1.61R$  are classified as medium entropy and alloys with  $\Delta S_{\text{mix}} \geq 1.61R$  are considered high entropy. It is easy to notice that these ranges correspond to the ranges  $N \leq 2$ ;  $N = 3-4$ ;  $N \geq 5$ .

$\Delta H_{\text{mix}}$  of HEAs can be defined through the following expression:

$$\Delta H_{\text{mix}} = \sum_{i,j} \Omega_{ij} c_i c_j \quad (3)$$

where  $\Omega_{ij} = 4\Delta H_{\text{mix}}^{\text{AB}}$  and  $4\Delta H_{\text{mix}}^{\text{AB}}$  are mixing enthalpies of binary A-B alloys.

The HEAs form simple solid solution phases when  $-22 \leq \Delta H_{\text{mix}} \leq 7$  kJ/mol, and  $11 \leq \Delta S_{\text{mix}} \leq 19.5$  kJ/mol.

The *severe lattice distortion effect* – a random distribution of atoms with different sizes in the crystal lattice – leads to distortion and affects the mechanical, transport and thermal properties of the material. To maintain the conditions for solid solution formation in HEAs, a difference in atomic radii ( $\delta$ ) calculated by using equation (4) should not exceed  $\delta \leq 8.5$  %.

$$\delta\% = 100\% \sqrt{\sum_{i=1}^n C_i \left(1 - \frac{r_i}{\sum_{j=1}^n C_j r_j}\right)^2} \quad (4)$$

Here,  $C_i$  and  $r_i$  denote the atomic fraction and atomic radius of the  $i^{\text{th}}$  element respectively.

A schematic view of the proposed localized lattice distortion effect in HEAs is presented in Fig. 1a, where

distortions arise not only from atomic size misfit, but also from differences in the crystal structure and bonding of alloying elements.

HEAs exhibit *sluggish diffusion kinetics* since atomic diffusion is more difficult through solid solutions with high concentrations of many elements. This effect allows for the formation of ultrafine crystallites and even amorphous phases during solidification, resulting in lower rates of nucleation and growth.

The complexity of HEA compositions gives rise to what are known as ‘cocktail effects’. It is difficult to determine precisely what is meant by this phenomenon and what makes it particularly special. The ‘cocktail effect’ can be described as the synergistic effect of the high entropy, sluggish diffusion and severe lattice distortion core effects in defining and affecting the overall properties of HEAs.

In the design of HEAs, it is always useful to predict possible phases and microstructures before conducting experiments. For this, the *valence electron concentration* (VEC) is an important factor for the formation of *fcc*, *bcc* or *hcp* structures in HEAs. For  $\text{VEC} < 6.87$  and  $\text{VEC} \geq 8.0$ , *bcc* and *fcc* phases are formed respectively, and both phases coexist for  $6.87 \leq \text{VEC} \leq 8.0$ . The  $\text{VEC} \leq 3$  favours the stability of *hcp* phase. The newly developed HEAs have garnered significant attention and research interest in various fields (e.g. materials science and biomedical engineering, electronics and optics) due to their unique concentrated solid solution structures and outstanding properties which cannot be obtained from conventional alloys. These include a combined increase in strength and ductility, high thermal stability and high hardness, great fatigue resistance, corrosion and wear resistance, and improved mechanical properties at cryogenic temperatures.

Most of the reported work on HEAs has focused mainly on microstructure, phase formation and

mechanical behaviour. Only recently magnetic properties of HEAs have been discussed in the literature [3] as excellent candidates for soft magnetic materials (SMM) with high saturation magnetization, high electrical resistivity to suppress the eddy-current loss, and malleability to facilitate processing and reduce processing costs. Such SMMs can be used in electrical power generation and transmission, electric motors, electromagnets and sensors, i.e. electromobility. Conventional alloys, on the other hand, have certain limitations, such as low electrical resistivity in Fe-Ni alloys or high brittleness in high-silicon steels at room temperature. Metallic glasses (metals with amorphous structure) are relatively unstable, and their sizes are limited by their glass-forming ability. Furthermore, HEAs have shown excellent potential for use as magnetocaloric materials for magnetic refrigeration applications. Also, hard magnetic properties have been found in FeCoNiAlCu<sub>x</sub>Ti<sub>x</sub>-based HEAs with coercivity  $H_c = 85784.5$  A/m and a maximum energy product ( $BH_{max}$ ) of 2.06 MGOe, slightly better than that of isotropically cast Alnico magnets.

Indeed, the magnetic properties of HEAs have a strong correlation with the phase constitution and their structure. However, a good understanding of the influence of phase composition and structural features on the magnetic properties of HEAs is still lacking.

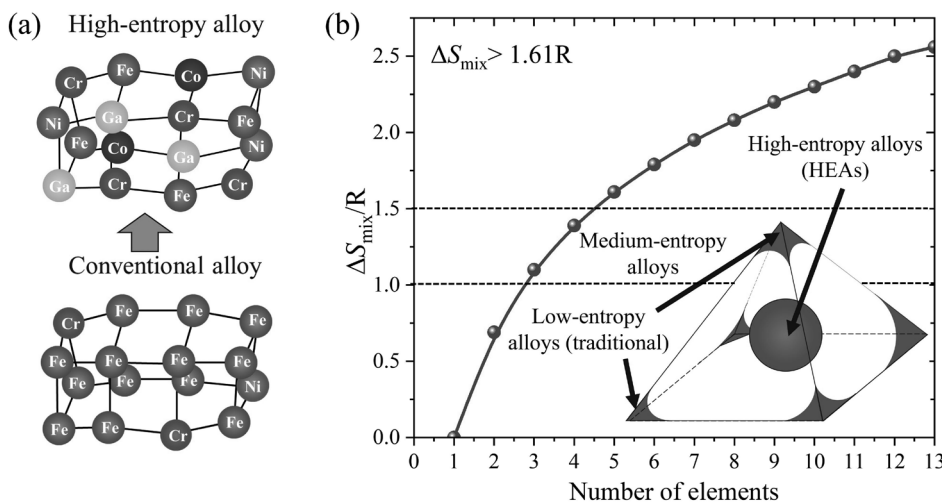
Studies of the magnetic properties of HEAs mainly focused on alloys composed of Co, Cr, Fe, Mn, Ni, Al, Cu and Ti. These alloys usually contain more than 50 at% magnetic elements – Fe, Co, Ni. For example, the ternary equiatomic FeCoNi with a single *fcc* structure exhibits high saturation magnetization ( $M_s$ ) (151 Am<sup>2</sup>/kg) with low coercivity  $H_c = 120.96$  A/m. Any addition of other paramagnetic or diamagnetic elements affects its magnetism. For example, the addition of Cr to a FeNiCo-based alloy significantly reduces the magnetization due to antiparallel coupling of the Cr spins to the Co/Fe/Ni spins and drives the CoCrFeNi alloy toward paramagnetism at room temperature ( $T_c$  near 100 K). The addition of Mn to the quaternary CoCrFeNi alloy does not result in significant structural and magnetic changes in the quintenary

CoCrFeNiMn composition (which is a classic example of an HEA, known as a ‘Cantor alloy’). It exhibits paramagnetic behaviour at room temperature (RT), despite the fact that it has a VEC = 8, which corresponds to the element Fe.

Replacing Mn with a non-magnetic element such as Ga to form equiatomic CrFeCoNiGa [3] results in a dramatic change from para- to ferromagnetism at RT. The data in the literature show that production from elemental powders resulted in the coexistence of *fcc* and *bcc* phases and an increase of the base alloy FeCoNiCr of  $M_s = 0.5$  Am<sup>2</sup>/kg (at 2 T) and  $T_c = 104$  K to  $M_s = 38$  Am<sup>2</sup>/kg and  $T_c = 703$  K for FeCoNiCrGa.

The mechanism behind the Ga-induced magnetic ordering is of particular interest due to the fact that Ga is a non-magnetic metal, and it offers the potential to design and optimize magnetic materials based on HEAs in the future.

So far, very few investigations devoted to this question have been presented, which may be attributed to the complexity of the problem related to the chemical and magnetic disorder present in HEAs.



(1) (a) Schematic crystal structures of conventional and high-entropy alloys. (b) The entropy of mixing of equiatomic alloys as a function of the number of the elements. The inset illustrates the difference between conventional and high-entropy alloys on a typical multicomponent phase diagram. Source: own illustration

Several processing routes (melting and casting, powder metallurgy, and deposition techniques) are used to prepare HEAs, which can produce distinct microstructures for the same composition, resulting in different properties. However, most of them are produced via melting routes. The common problem of the conventional melting route is the non-homogeneous microstructure in the solidified product. Therefore, it is essential to do the annealing at a higher temperature followed by quenching to obtain a homogeneous structure. The grain size usually reaches several hundred micrometers with arc melting followed by casting. Also, control of the sublimation and evaporation of low-boiling elements has proved difficult, thus making compositional control more difficult.

On the other hand, high-energy ball milling (HEBM) is a well-known solid state, non-equilibrium and promising approach for producing homogeneous microstructures in a huge variety of materials, such as intermetallics, ordered compounds, solid-solution alloys, amorphous structures (metallic glasses), quasi-crystalline phases and nanocomposites. HEBM also takes advantage of the extended solubility of solids even in immiscible systems [4]. Compared to conventional mechanical alloying, short-term HEBM provides a much higher energy input and reduction of contamination from milling balls/jars. Nanocrystallinity adds another exciting aspect to the huge compositional space and complex dimensions of HEAs. Nanostructured HEAs have shown improved mechanical properties, excellent magnetic behaviour and high thermal stability [5].

Since the first reports of a nanocrystalline AlCrCuFeTiZn alloy in 2008, several reports on mechanically alloyed nanocrystalline HEAs have been published. Most of the solid solution phases in HEAs formed within 15–40 hours of conventional milling. Such prolonged milling hours often result in amor-

phous HEAs. For example, the complete amorphization of AlBCFeNiSi and AlBCeFeNiSi alloys takes 140 h and 240 h of milling, respectively. The disadvantage of the long milling duration during mechanical alloying could be overcome by using short-term high-energy ball milling [6]. In comparison to the problem with low melting Ga in an arc melting process, HEBM offers a novel, fast synthesis approach for this type of material [3].

In the present work, we show the design possibility of new magnetic nanocrystalline CoCrFeNiGa high-entropy alloy powder particles prepared by HEBM, with the subsequent consolidation into bulk materials by spark plasma sintering (SPS). Evolution of phases, microstructure and magnetic properties have been investigated.

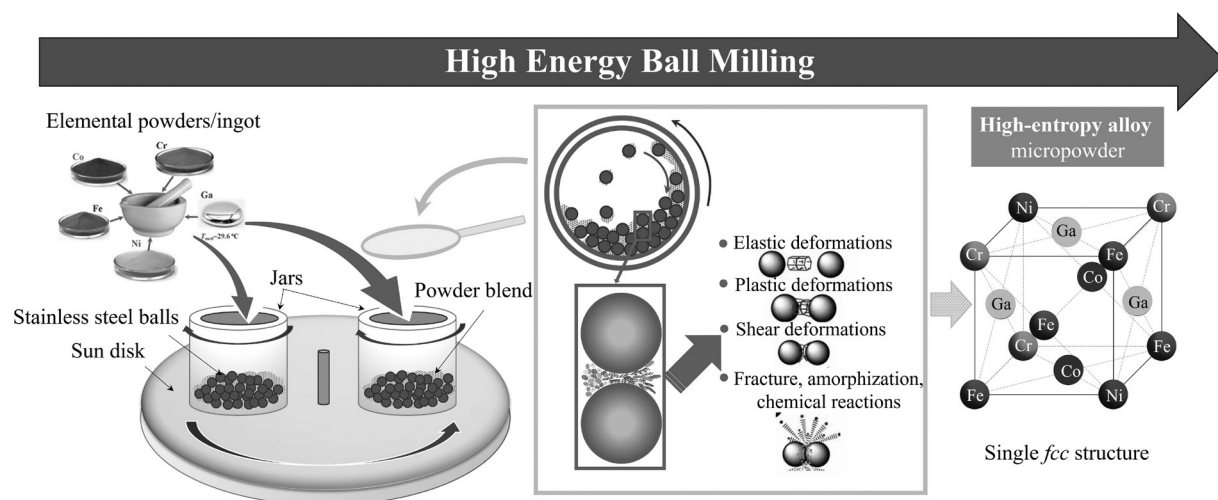
During the HEBM process, pure metal powders are mixed with a certain proportion and put into a planetary ball mill together with a milling medium (stainless steel balls) (Fig. 2). Subjected to the strong collision and agitation of the steel balls, different metal powders undergo repeated cold welding, milling and rewelding. Under high-speed friction, repeated cold welding induces the bonding of metal atoms between the contact surfaces of different metal powders. In addition, smaller particles with lattice defects are constantly generated because of repeated impact and breaking during the milling process. At the same time, the heat generated locally at the instant of the collision promotes the diffusion of lattice defects, contributing to the formation of interatomic bonds. As a consequence, HEA powders are able to form.

By using short-term HEBM, we can provide much higher energy input to the composition and synthesize homogeneous HEA with different stable and metastable structures.

Let's take a look at the synthesis of CoCrFeNiGa HEA powder particles in detail. The HEBM processing leads to significant structural changes in the composition. After

only 15 minutes of HEBM, a gradual broadening and partial overlapping of Co, Cr, Fe and Ni diffraction peaks accompanied by a decrease in their intensity has been observed (Fig. 3k). When the HEBM time reaches 45 minutes, (111), (200) and (220) peaks of the *fcc* phase appear, while Bragg peaks of individual elements have lost their identity completely. As a result, the formation of two solid solutions with *bcc* and *fcc* structures has been detected (Fig. 3k). Further mechanical treatment leads to a gradual *bcc* → *fcc* transformation together with partial disordering in CoCrFeNiGa alloy powders due to intense mechanical deformation. After 190 minutes of HEBM, (110), (200) and (200) peaks of a *bcc* phase are not pronounced. Finally, CoCrFeNiGa HEA powder with a single *fcc* structure with a crystallite size of 10 nm is formed. The microstructural transformation of the CoCrFeNiGa powder blend as a function of HEBM time was studied by scanning electron microscopy (SEM) analysis in a back-scattered electron (BSE) mode, which provides information about the composition and atomic number contrast of the sample.

Figures (3a–j) show a set of SEM images taken from metallographic cross-sections of CoCrFeNiGa samples compacted from different composite powder particles produced via HEBM with different processing times ( $t = 30$ –190 min). Short mechanical treatment ( $t = 30$  min), leads to the formation of coarse oval agglomerates ~100–200  $\mu\text{m}$  in size with a layered structure due to multiple flattening and adhesion of ductile metals between them (Figs. 3a, b). Further mechanical treatment ( $t = 60$ –90 min) leads to a decrease in the average layer thickness, and homogenized areas were also detected (Figs. 3c–f). At  $t = 180$  min, the layer structure vanished completely, and a relatively uniform distribution of initial elements was observed (Figs. 3g, h). An additional milling of the CoCrFeNiGa powder blend



(2) A schematic diagram of HEBM processing for the synthesis of HEA microparticles: schematic view of ball mill (left); schematic view of the motion of the ball and powder mixture including different possible impacts on materials between colliding balls during milling, *fcc* crystal structure of the HEA powder.

Source: own illustration

for 10 minutes in  $C_3H_7OH$  was allowed to get an excellent compositional homogeneity of HEA particles. This was inferred from the SEM BSE of Figures (3i, 3j) where the contrast is defined by the atomic number of a given phase (contrast is Z-sensitive): for example, the Cr-rich areas ( $Z = 24$ ) look darker than the Ga-rich ones ( $Z = 31$ ). Moreover, the uniform distribution of principal elements was also confirmed by energy-dispersive X-ray spectroscopy (EDX).

HEA powders obtained by HEBM need to be sintered to achieve bulk material. While conventional sintering of nanocrystalline alloy powders for a long duration can lead to significant grain growth, SPS involves sintering for a very short time (Fig. 4a). This helps to retain the nanostructures in the mechanically alloyed powder.

The HEBM CoCrFeNiGa HEA powder was SPS-consolidated in vacuum in a Labox 650 facility (Sinter Land, Japan). The powder mixture was placed in a cylindrical graphite die (inner  $\varnothing = 12.7$  mm) and uniaxially compressed at 10–50 MPa. The sample was heated at a rate of 100 K/min to 1073 K by

passing rectangular pulses of electric current through the sample (schematic view in Fig. 4a). The dwell time at sintering temperature was just 10 min. SPS-produced disks were 2–3 mm thick and 12.7 mm in diameter compacts (Fig. 4a).

The use of HEBM-produced CoCrFeNiGa powders enabled the synthesis of a homogeneous bulk HEA [3], which was not possible starting with elemental powders of Co, Cr, Fe, Ni and Ga ingots in a direct SPS process.

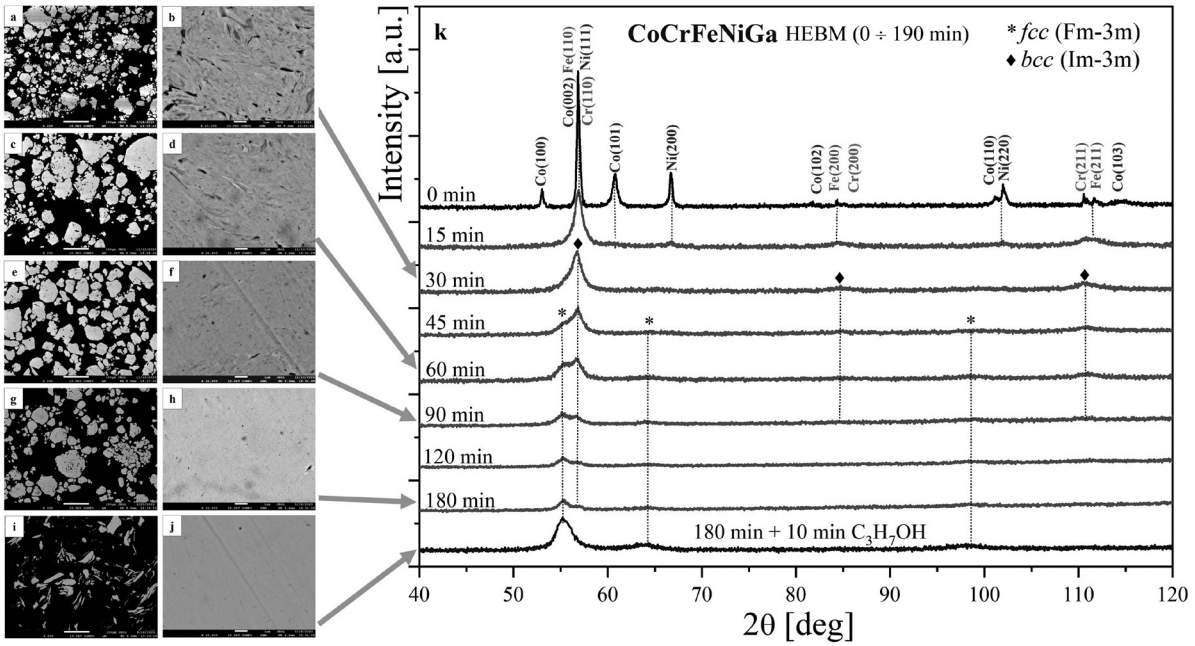
SPS consolidation of the equiatomic CoCrFeNiGa HEA powder (Fig. 4b) at 1073 K led to partial decomposition of the *fcc* structure. XRD data showed that SPS-consolidated material (Fig. 4b) contained a mixture of *bcc* and *fcc* phases.

Recently, we demonstrated [3] that an HEA bulk sample produced by the combined use of HEBM and SPS has a high microhardness (6.343 GPa), which is five times more than that of the bulk prepared from elemental powders, and even higher than those observed in the CoMnFeNiCr (Cantor) alloy. The magnetic properties of the CoCrFeNiGa HEA powder and HEA bulk sample were determined

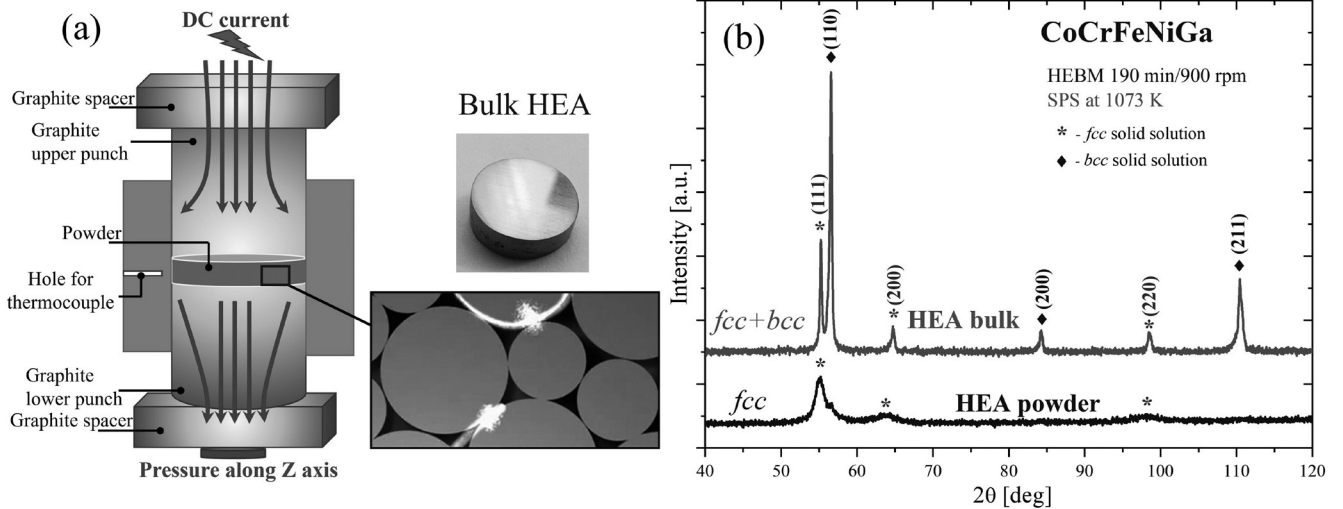
using a Quantum Design DynaCool physical property measurement system (PPMS) at various temperatures under external magnetic fields of up to 9 T.

A big difference is immediately apparent in the temperature-dependent magnetization  $M(T)$  curves for the CoCrFeNiGa HEA powder and the bulk sample (Fig. 5a). The HEA powder has (at 5K) a magnetization of about 32 Am<sup>2</sup>/kg (about half of that of Ni), while the bulk HEA has a slightly higher value of 39 Am<sup>2</sup>/kg. This may be due to a slight change in composition during the sintering. When the temperature is increased to 390 K, the magnetization of the HEA powder decreases to 2 Am<sup>2</sup>/kg, which can be attributed to the almost complete loss of magnetic order with a Curie temperature  $T_c = 95$  K (determined as a first derivative from the  $M(T)$  curve measured at 100mT). For the bulk HEA, a higher temperature was required to determine the ferro-to-paramagnetic transition (i.e.  $T_c$ ).

SPS consolidation led to the enhancement of magnetic properties for the equiatomic CoCrFeNiGa HEA powder and increased the  $T_c$  to 750–905 K. Field-dependent magnetization measured at 300 K (i.e. RT)



(3) SEM images (cross-section) of CoCrFeNiGa powder blends as a function of HEBM time at 900/1800 rpm: (a) and (f): 30 minutes; (b) and (g): 60 minutes; (c) and (h): 90 minutes; (d) and (i): 180 minutes; (e) and (j): 180 + 10 minutes. Scale bar for (a)–(e) is 100  $\mu\text{m}$ , for (f)–(j) is 1  $\mu\text{m}$ . (k) XRD patterns of CoCrFeNiGa powder mixtures taken after different time periods  $t$  of HEBM (inserted to the left) at 900/1800 rpm. Source: own illustration



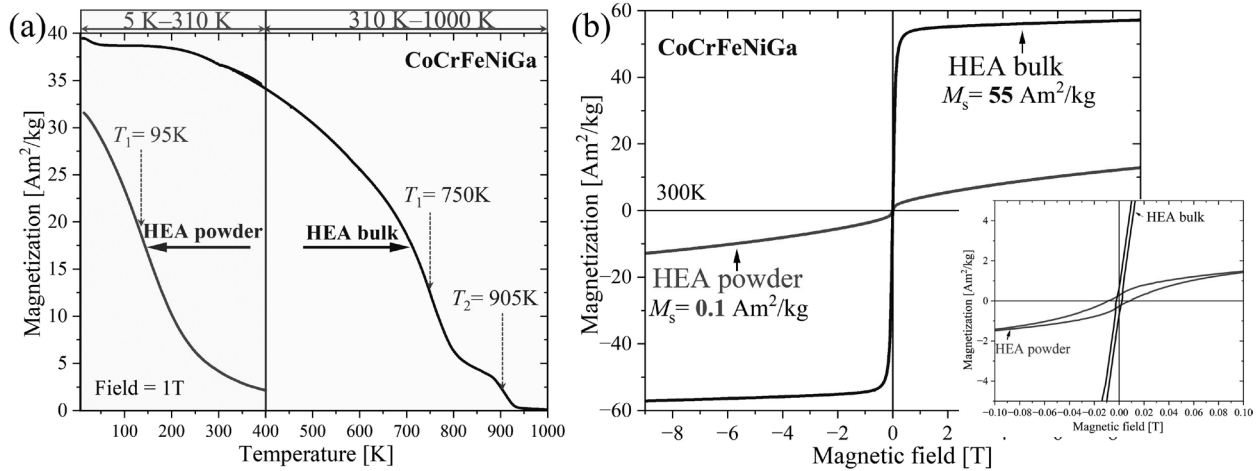
(4) (a) Schematic view of spark plasma sintering (SPS); (b) XRD patterns of HEA powder before and after SPS consolidation. Source: own illustration

shows paramagnetic behaviour for the HEA powder with a saturation magnetization  $M_s = 0.1 \text{ Am}^2/\text{kg}$ , while the bulk HEA is ferromagnetic with  $M_s = 55 \text{ Am}^2/\text{kg}$  (Fig. 5b).

### Zusammenfassung

Zusammenfassend lässt sich sagen, dass das HEBM-Verfahren für HEA einen vielversprechenden neuen Weg für homogene HEA-Partikel

darstellt, die als Vorläufer für die Herstellung homogener HEA-Materialien durch Spark-Plasma-Sintern dienen. Der schnelle HEBM-Produktionsprozess ermöglicht ein „Upscaling“, um Pulver in Mengen zu erhalten, die auch in der additiven Fertigung verwendet werden können.



(5) Magnetometry data of CoCrFeNiGa HEA powder and HEA bulk sample: (a) temperature-dependent magnetization  $M(T)$  recorded at 1T. (b) Field-dependent magnetization  $M(H)$  measured at 300 K. Source: own illustration

Die Untersuchung der magnetischen Eigenschaften in Abhängigkeit von der Mikrostruktur und der Zusammensetzung steckt noch in den Kinderschuhen, insbesondere im Hinblick auf die Rolle, die verschiedene Verarbeitungsparameter bei der Steuerung der Gesamtmagnetisierung, der Curie-Temperatur und der magnetischen Anisotropie, die mit der magnetischen Koerzitivfeldstärke zusammenhängt, spielen. Folglich sind weitere Untersuchungen erforderlich, um die magnetischen Parameter zu verbessern und abzustimmen. Wie oben gezeigt, spielen die Synthesebedingungen eine entscheidende Rolle, und eine systematische Untersuchung der HEBM-Verarbeitungszeit, des Magnetismus, der Mikrostruktur und der chemischen Homogenität ist erforderlich und wird fortgesetzt werden.

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*Annotations*

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**Natalia Shkodich** graduated from Kazan State Technological University (Kazan, Russia) in 2006 with a Master Degree in the field of chemical engineering & technology. She got her degree of Cand. Sci. (Phys.-Math.) – Russian analog of PhD – from the Russian Academy of Sciences in 2011. In the period from 2005–2021 (Junior researcher–Senior researcher) she worked at the Laboratory for dynamics of micro heterogeneous processes, Merzhanov Institute of Structural Macrokinetics and Materials Science, Russian Academy of Sciences, in the field of materials science, mechanical activation/alloying, powder metallurgy, nanotechnology, consolidation processes (spark plasma sintering, hot pressing), and combustion synthesis. Earlier, she received her research practice, experience, and skills at the following in-

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