Nanocrystalline FeCr alloys synthesised by severe plastic deformation – A potential material for exchange bias and enhanced magnetostriction

Lukas Weissitsch*, Stefan Wurster, Alexander Paulischin, Martin Stückler, Reinhard Pippan, Andrea Bachmaier

Erich Schmid Institute of Materials Science of the Austrian Academy of Sciences, 8700 Leoben, Austria

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ABSTRACT

This work gives insights into processing and characterisation of bulk nanocrystalline FeCr materials. The investigated FeCr alloys, consisting of 30, 50 and 70 at.% ferromagnetic Fe and remaining anti-ferromagnetic Cr, are processed by arc melting and subsequent severe plastic deformation by high pressure torsion. The similarities between elemental Fe and Cr in combination with the nanocrystalline structure of the as-deformed alloys necessitates advanced characterisation techniques for the as-deformed state: In-situ annealing synchrotron X-ray diffraction measurements as well as electron microscopy experiments are linked to magnetostrictive measurements and reveal a single phase microstructure. Surprisingly, the nanocrystalline FeCr alloys remain supersaturated solid solutions upon annealing above 500 °C, meaning a decomposition in a FeCr nanocomposite is suppressed. For the chosen annealing conditions grain growth is faster than decomposition and enhanced magnetostrictive values are found compared to materials in the as-deformed state.

1. Introduction

Tuning material properties by forming single phase alloys and varying chemical composition is often impeded by the existence of large miscibility gaps at the thermodynamic equilibrium of binary phase diagrams. Notwithstanding the difficulties in fabrication, there is a strong interest in such metastable phases created materials as they offer the opportunity to tune and enhance physical properties. For quite some time, it is known to overcome these processing limits by techniques such as vapour deposition [1], mechanical alloying (MA) [2] or severe plastic deformation (SPD) generating metastable phases [3–5]. The formation of supersaturated solid solutions by MA or SPD is accompanied by a high defect density and decreased microstructural sizes. This evinces characterisation challenges for the binary nanocrystalline FeCr system: Due to the same crystal structure and similar lattice parameters of elemental Fe and Cr, it is difficult to distinguish whether finely dispersed elemental nanocrystalline grains or a (supersaturated) solid solution is present [2]. Possible characterisation methods are magnetic measurements. Apart from the tuneability of the saturation magnetostriction of heterostructures [6,7] Bormio-Nunes et al. [8] reported an increasing saturation magnetostriction value for FeCr solid solutions. The aforementioned heterostructure of coexisting ferromagnetic Fe and anti-ferromagnetic Cr phases could give rise to an effect referred to as exchange bias.

The exchange bias features a shifted hysteresis loop, which originates from an unidirectional anisotropy created at the interface of the two types of magnetic materials. This occurs, when the ferro-antiferromagnetic phase boundary is cooled through the Néel temperature (\(T_N\)) while a magnetic field is applied [9]. Since magnetic exchange interactions appear on the nanometre-scale and the exchange bias effect is related to the phase boundaries, this effect is typically observed in microstructures obtained by well controllable synthesis techniques. Thin films or core–shell nanostructures [10] are well controllable during processing and therefore, such material systems allow a proper investigation of fundamental physical properties [11]. On the other hand, it is difficult to investigate the same effects in bulk materials [12], exhibiting dimensions of several millimetres or larger. The bottleneck nowadays is the synthesis of a nanostructured bulk material exhibiting a high homogeneity therefore, enabling an upscaling of materials with improved magnetic properties. Notwithstanding the above, the amount of interfaces is thought to directly influence the intensity of the magnetic effect, which makes magnetic nanostructured bulk materials necessary [9,13].

Severe plastic deformation by high-pressure torsion (HPT) is a
promising approach to overcome processing limitations, for above
mentioned miscibility gaps of binary material systems [5] and further-
more a method of choice to tailor magnetic properties [14,15]. One big
advantage of materials processed by HPT is the sample size, as they
exhibit bulk dimensions up to several centimetres [16,17]. The principal
goal of this work is the production of a bulk material consisting of
alternating interfaces of elemental ferromagnetic Fe and anti-
ferromagnetic Cr. Therefore, the synthesis of a nanocrystalline super-
saturated solid solution of FeCr by HPT is aimed for a subsequent
annealing treatment. The increased total grain boundary area of nano-
crystalline materials is expected to favour a decomposition [18] of the
FeCr solid solution in separated phases and therefore, into a nano-
structured composite. A following magnetic field cooling is meant to
induce an exchange bias, as already reported for HPT-compacted Co-
NiO heterostructures [19]. However, processing and the characteriza-
tion of nanocrystalline HPT deformed FeCr-alloys prior to field cooling is
challenging. Evading processing limitations due to a pronounced
miscibility gap in the binary phase diagram [20], we already reported an
enormous increase in hardness during HPT-deformation but also on the
successful processing and formation of a homogeneous nanocrystalline
microstructure for a wide range of chemical compositions [3]. In this
work we clarify, if these materials exhibit a supersaturated solid solution
or if they are already in a nanocrystalline two phase state. We report on
in situ high-energy X-ray diffraction (HEXRD) experiments where a solid
solution without any decomposition upon annealing is presumed and
verify these results by magnetostriective measurements.

2. Experimental

Binary alloys consisting of nominal 30, 50 and 70 at.\% Fe and 70, 50,
and 30 at.\% Cr, respectively, were processed by arc melting and sub-
sequent HPT-deformation. As starting materials, conventional flakes
(Fe: 99.99+\%<10 mm, Cr: 99.995% 1-25 mm, both from HMW Hauner
GmbH & Co. KG) were used. All materials were stored and handled in an
Ar-filled glovebox to protect the high purity materials against
contamination.

For arc-melting (AM/0.5 device, Edmund Buehler GmbH), a Ti-
generated high purity Ar atmosphere was used. To ensure chemical ho-
mogeneity the ingots were turned around and remelted at least five
times. From arc melted ingots, cylinders with 8 mm diameter were
prepared by electrical discharge machining and cut in discs with 1 mm
thickness, which were subsequently deformed by HPT. A detailed
description of the used HPT setup can be found in Ref. [21]. A nominal
pressure of 7.5 GPa with a rotational frequency of 0.6 min^{-1} was used
for the deformation process and 21 to 25 revolutions are applied (cor-
responding to a shear strain γ of ~ 450 at radius \( r = 3 \, \text{mm} \) [22]). To
avoid an increase of the processing temperature during deformation,
high pressure air cooling was conducted.

After deformation, the sample is cut for further investigations and all
measurements were performed at a radius \( r \geq 2 \, \text{mm} \). Respective ori-
entations are explained in detail in Reference [15]. Scanning electron
microscopy (SEM; LEO 1525, Carl Zeiss Microscopy GmbH) images were
made in tangential HPT-disc orientation in backscattered electron (BSE)
detection mode. The chemical composition was determined by energy
dispersive X-ray spectroscopy (EDX; XFlash 6/60 device, Bruker), using
the Bruker software package Esprit 2.2. The grain size of selected sam-
ple was quantitatively evaluated by Transmission Kikuchi Diffraction
(TKD) and electron backscatter diffraction (EBSD) measurements using a
Bruker e-Flash system detector. Hardness measurements (Micromet 5104,
Buehler) were performed in tangential direction on the polished sample
along the diameter in steps of \( \Delta r = 0.25 \, \text{mm} \) and were averaged for a
radius 2 mm \( r < 3.5 \, \text{mm} \). For annealing experiments, quarters or half
discs of as-deformed samples were exposed to 300 °C, 400 °C and 500 °C
for 1 h in a conventional furnace. The samples were wrapped in a pro-
tective foil before heat treatment and were quenched with ethanol after
the annealing process.

For magnetostriective measurements, an electromagnet (Type B-E 30,
Bruker) with conical poles (diameter 176 mm), providing a constant air
gap (50 mm) and a maximal field of 2.25 T, was used. Within the air gap,
the sample was placed into an encapsulating sample chamber, protect-
ning from rapid temperature changes. The magnetostriiction was
measured using strain gauges (HBM, 1-LY11-0.6/120) applied with glue
Z70 from HBH. As a reference a piece of W, a very low magnetostriective
material is measured at the same time with a strain gauge of the same
batch. According to [23], the magnetostriiction of W can be expected to
be below 0.005 ppm in a magnetic field of 2 T. The strain gauge on the
sample and the reference were placed parallel and on top of each other
within the sample chamber and conducted to a half-Wheatstone bridge.
The signal of the strain gauges was processed with an amplifier (HBM-
Quantum® MX411). Additionally, the constancy of the temperature was
confirmed by a temperature measurement within the sample holder and
the applied magnetic field was recorded, using a Hall-probe (Model 475
DSP, Lakeshore).

Synchrotron high-energy X-ray diffraction (HEXRD) experiments
were carried out at the beamline P21.2 at PETRA III (DESY, Hamburg,
Germany). The specimens were measured in transmission mode, while
the spot size was set to 200 × 200 μm² and a photon energy of 60 keV
was used. In-situ annealing HEXRD experiments were performed using
the following sequence: Heating to 520 °C, holding the temperature for
1 h and cooling. A Varex XRD 4343 flat panel detector captured
diffraction patterns every 5 s. The experimental setup was realised by a
heating microscope stage (THMS600 Linkam, Tadworth, United
Kingdom), which was adapted to place the samples inside an Ar-flushed
chamber to suppress the formation of oxides. The custom made chamber
is designed to minimize the interaction of the beam with the furnace
and allowed the diffracted beam to exit the probe chamber while the tem-
perature was measured via a thermocouple. The temperature was cali-
brated by measuring a Cu sample with the same settings and comparing
the changing lattice parameter during thermal expansion. For data
processing, the pyFAI package [24] and further analysing the Matlab
voigt package [25] was used.

3. Results

3.1. Characterisation of the microstructure

The microstructures of room temperature (RT) HPT-deformed sam-
ple at \( r = 3 \, \text{mm} \) with different chemical compositions are shown in
Fig. 1. The Fe-content increases from the top to bottom row. In the first
column, BSE images of as-deformed FeCr alloys are depicted. Nano-
crystalline and homogeneously deformed microstructures are visible.
To quantify the grain sizes of the alloys in the as-deformed state, TKD
is conducted exemplarily for one composition (Fe₉₀Cr₁₀). In Fig. 2 a)
a crystal orientation map revealed by TKD is shown. Recording several
images and interpolating the data of 1256 grains with a log-normal
distribution reveal a median grain size of 101 nm (standard deviation
= 86 nm; \( \sigma = 0.579, \mu = -2.288 \)). Please note the different scale bar in
Fig. 2 b), where an EBSD scan of the same alloy after annealing for 1 h at
500 °C is shown. The obtained median grain size is 307 nm (1456 grains,
standard deviation = 160 nm; \( \sigma = 0.449, \mu = -1.180 \)). Results from
Vickers-hardness testing are indicated as insets within each SEM
micrograph (Fig. 1). The hardness measurements along the radius,
display a constant hardness, which indicates a homogeneously deformed
state (denoted as ‘as-deformed’ in the following). To induce decompo-
sition, the as-deformed samples are annealed for one hour at different
temperatures (Fig. 1). The change in the microstructure strongly de-
pends on the chemical composition. Nearly no change in the SEM images
for the Cr-rich sample (Fe₈₀Cr₂₀), depicted in the first row of Fig. 1, is
visible, when annealing up to 400 °C. However, the hardness increases,
which is a common feature for nanocrystalline materials [26–28], is
covered. A slightly increased grain size, accompanied by a hardness
decrease, is seen for the 500 °C annealed sample. The medium
composition sample (Fe$_{50}$Cr$_{50}$) shows a slight contrast difference in SEM images between the as-deformed and 300 °C annealed state. The microstructure remains almost constant even when annealing at 400 °C (shown in the 3rd column) until a significant grain growth is found for 500 °C annealing. The hardness increases upon annealing at 300 °C but decreases for higher annealing temperatures. Differences for every state are visible for the Fe rich sample (Fe$_{70}$Cr$_{30}$). At 300 °C, a slight grain growth is observed together with an increased hardness. Samples annealed at 400 °C and 500 °C exhibit a significant increase in grain size as well as a decrease of microhardness below the as-deformed state. Summarized, at intermediate temperatures the hardness increases for all chemical compositions, whereby the hardness peaks at higher temperatures for higher Cr content. Further, microstructural changes and grain growth are stronger pronounced for FeCr alloys containing a higher amount of Fe. Laboratory XRD measurements of the as-deformed and annealed states (not shown) revealed broad peaks. They can either result from the formation of a supersaturated state or be due to the nanocrystalline state of individual Fe and Cr phases present in the as-deformed condition. Additionally, a co-existence of both configurations is conceivable. Thus, to clarify this point and to further investigate the decomposition of FeCr alloys, which are possibly in a non-equilibrium state, in situ annealing experiments using synchrotron XRD are performed. Due to the high brilliance of the beamline [29], it was expected to resolve individual peaks for Fe or Cr phases, if existent, and to precisely determine the phases of the as-deformed and annealed states. Representatively, the most intense peak [110] of selected patterns of the in situ experiment is shown in Fig. 3 for all three FeCr compositions. The vertical lines indicate reference values for elemental Fe (solid, turquoise) and Cr (dashed, brown) calculated with lattice constants from [30]. The depicted peaks correspond to different temperatures during the in situ experiment. The top peaks (red) belong to

![BSE images of the microstructure of HPT-deformed FeCr alloys. The Fe content increases from the top to the bottom row. The first column shows the microstructure of as-deformed samples. The next columns depict microstructural changes with increasing annealing temperatures (300 °C, 400 °C and 500 °C, respectively). Averaged Vickers-hardness values are depicted in the corresponding images as insets. The magnification is the same in all images.](image1)

![Crystal orientation maps of a) an as-deformed Fe$_{50}$Cr$_{50}$ alloy recorded in axial HPT disc direction by TKD. b) EBSD scan of an Fe$_{50}$Cr$_{50}$ alloy after annealing at 500 °C (1 h) measured in tangential direction but showing the crystal orientation in axial direction. Note the different scale bars.](image2)

![HEXRD synchrotron data of the [110] peak for HPT-deformed FeCr samples consisting of a) Fe$_{30}$Cr$_{70}$, b) Fe$_{50}$Cr$_{50}$ and c) Fe$_{70}$Cr$_{30}$. Vertical lines denote theoretical reference lines for Fe (solid, turquoise) and Cr (dashed, brown). The selected patterns belong to in situ annealing experiments and are extracted at different times and temperatures, visualized d) by arrows in an exemplarily temperature profile.](image3)
the beginning of the experiment, representing the as-deformed state. A significant peak broadening is again found for all three chemical compositions. According to Vegard’s law [31] the peak positions follow the trend of the chemical composition determined by EDX measurements of the FeCr alloys. The Fe contents obtained with EDX for the alloys depicted in Fig. 3(a)–(c) are 27.8 ± 3.3 at.%, 48.7 ± 0.8 at.% and 69.1 ± 1.1 at.%, respectively. The blue peaks are recorded at the end of the heating process, when a constant temperature of 520 °C is reached. Because of the thermal expansion of the lattice, a shift towards a lower reciprocal lattice vector is visible. This is accompanied with a significantly sharpened shape of the peak, indicating larger crystallite sizes and/or diminishing crystallographic defects. The green peak is recorded after 1 h of isothermal annealing at 520 °C and shows almost no deviations with respect to the blue pattern. The purple peaks at the bottom correspond to an ex-situ measurement after the sample cooled down to room temperature and can be directly compared with the red line. The recorded patterns are fitted with Voigt-profiles. It should be mentioned, that for every peak one single Voigt-profile is used. Although fitting by one peak implies a solid solution, an overlapping of two peaks corresponding to separated Fe and Cr is also possible. Unfortunately, the similar crystal structures (both body centred cubic) and the similar lattice parameters with less than 0.02 Å difference (see Reference [30]) lead to very similar peaks for Fe and Cr, with almost no differences. Despite the high brilliance of the synchrotron, a pattern fitting with two separated Voigt-peaks is ambiguous. The shape, as well as intensity and position, strongly depend on the fitting starting parameters which allows no clear statement. Nevertheless, using a single Voigt peak profile, a high accordance is reached for the calculated pattern with respect to the measured HEXRD data. The peak positions as a function of the temperature process while heating (filled symbols) and cooling (empty symbols) is shown in Fig. 4. A difference between these processes is recognised: While the peak position changes as expected linearly during cooling, the heating curve is bended. Although, no peak separation is recognised: While the peak position changes as expected linearly during the temperature process while heating (full symbols) and cooling (empty symbols) the measured HEXRD data. The peak positions as a function of the reciprocal lattice vector is visible. This is accompanied with a significant peak broadening, respectively. The evolution of peak widths for each alloy is shown in the supplementary data file. It is found that the Cauchy peak width decreases with increasing annealing temperature. In contrast, the Gaussian FWHM barely changes until 250 °C before it decays rapidly with increasing temperature, indicating a reduction in internal stresses. The contribution of internal stresses to the overall peak width observed in Fig. 3 is strongly reduced, when the heating process is finished. The remaining narrowing of the peaks, upon further isothermal annealing, originates from size effects and therefore grain growth, which is in accordance with SEM investigations of the annealed FeCr alloys (see Fig. 1). After annealing for one hour at 520 °C, no peak separation or broadening of the peak due to a superposition of its elemental Fe and Cr peaks is found. We therefore conclude, that all FeCr alloys exhibit a nanocrystalline, supersaturated solid solution state before and after the annealing treatment. To decompose these alloys, higher annealing temperatures are necessary.

3.2. Magnetostrictive measurements

In principle, 3d-elements like Fe exhibit a low magnetostrictive response [33] but when alloyed with Al, Si, Ge or Ga an increased value was found [34]. This is explained by the substitution of an Fe atom by a non-magnetic metal. The magnetostriction of arc melted FeCr alloys without subsequent plastic deformation was measured by Bormio-Nunes et al. [8]. They processed FeCr solid solutions, with a Cr content between 5.3 and 30.7 at.% and found an increased saturation magnetostriction compared to the magnetostriction of the pure elements. Besides the interesting question, if a higher Cr content further increases the magnetostriction, an enhanced value confirms our results from Section 3.1, as the increased magnetostriction is only present in the FeCr solid solution. Therefore, we conducted magnetostrictive measurements on all FeCr alloys in the as-deformed state as well as in one annealed state.

Assuming a perfectly isotropic material, the magnetostriction measured parallel and perpendicular to the applied magnetic field [35], follows \( \lambda_\parallel = -2 \lambda_\perp \). The anisotropy of magnetostrictive behaviour is further influenced by the crystal structure and crystallographic directions. Therefore, the effective saturation magnetostriction \( \lambda_\parallel \) for polycrystalline materials is usually described by

\[
\lambda_\parallel = \frac{2}{3}(\lambda_{||} - \lambda_{\perp})
\]

According to Eq. (1) the magnetostrictive behaviour of as-deformed FeCr alloys as a function of the applied magnetic field is shown in Fig. 5. The saturation value increases with increasing Fe content. At a maximal applied field \( \mu_0 H = 2.25 \) T the signal is close to 0 ppm for the

![Fig. 4](image-url)  
Fig. 4. [110] peak position for every pattern during heating (full symbols) and cooling (empty symbols) for HPT-deformed FeCr alloys determined by fitting the HEXRD in situ data by a single Voigt profile. The cooling is not covered experimentally for the Fe30Cr50 alloy.

![Fig. 5](image-url)  
Fig. 5. Effective magnetostriction as a function of applied magnetic field for three FeCr alloy after HPT-deformation at RT.
as-deformed Fe$_{50}$Cr$_{70}$ sample, whereas the samples consisting of Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$ reach 8 ppm and 17 ppm, respectively. To give a comparison, the magnetostriction of elemental Cr is very small ($\lambda^m_{Cr} < 1$ ppm) [36] and elemental Fe exhibits a crystallographic anisotropic magnetostriction, which results in a negative value $\lambda^m_{Fe} = -9.3$ ppm according to Eq. (1) [35,37]. Preliminary SQUID magnetometry measurements conducted on the Fe$_{50}$Cr$_{50}$ sample, exclude a paramagnetic state at room temperature but indicate the presence of superparamagnetic particles, possible existing within the high amount of grain boundaries of the nanocrystalline material [38]. Applying these values and using the $\frac{f}{V}$ volume fraction $f$, the volumetrically weighted balance between a magnetostrictive $\lambda^m_{eff}$ and a non-magnetostrictive $\lambda^s_{eff}$ component for the effective saturation magnetisation $\lambda^s_{eff}$

$$\lambda^s_{eff} = f \lambda^m_{Fe} + (1-f) \lambda^m_{Cr}$$

(2)
gives deeper insights into the Fe based nanocrystalline alloys [39]. Obviously, this approximation does not hold for our presented alloys as Eq. (2) yields an even smaller quantity in $\lambda$. We can either conclude on an existing solid solution or must assume other mechanisms giving a large positive contribution to the measured magnetostriction. If we assume separated phases, Eq. (2) does not consider contributions of surface spins and grain boundaries. Nevertheless it is known, that both, surface- and bulk- magnetoelastic coupling contribute to the magnetoelastic tensor [6], which is further important for nanocrystalline materials [40]. To describe the magnetostrictive behaviour of nanocrystalline materials, Slawska-Waniewska et al. [41] expanded Eq. (2) to

$$\lambda^s_{eff} = f \lambda^m_{Fe} + (1-f) \lambda^m_{Cr} (f) + f \lambda^s_{eff}$$

(3)

where $\lambda^s_{eff}$ describes the interface contribution such as surface spins to the effective saturation magnetostriction. $\frac{f}{V}$ is the surface to volume fraction, which can be rewritten in $\frac{f}{V}$ for spherical grains with an effective grain radius $R$. For the FeCr system only the third term contribute with a positive value to $\lambda^s_{eff}$ keeping in mind, that the measured $\lambda_5$ values for Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$ presented in Fig. 5 are positive. If the second term is neglected and the measured values for $\lambda^s_{eff}$ as well as a grain size radius of 50 nm (median grain diameter obtained by TKD measurements - see Fig. 2) are used, we can estimate a value for $\lambda^s_{eff}$. For the Fe$_{70}$Cr$_{30}$ composition a $\lambda^s_{eff} \approx 400 \times 10^{-6}$ nm is necessary to fit our measurements. Comparing $\lambda^s_{eff}$ values for FeCr alloys to other Fe based nanocrystalline materials, they differ by one (Fe$_{50}$Cr$_{50}$) or two (Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$) orders of magnitude [40,41]. Therefore, surface spin contributions to the effective saturation magnetostriction are not excluded but cannot be the main contribution of the increased $\lambda^s_{eff}$ and consequently, Fe and Cr have to be present in the form of a solid solution.

Hall et al. [42] used etched single crystals of FeCr and found an increasing magnetostriction ($\lambda_{100} = 51.7$ ppm) when alloying Fe with Cr up to 21.1 at.% for the crystallographic [100] direction. But they also describe a concurrently decreased value for the [111] direction ($\lambda_{111} = -2.7$ ppm), if compared to elemental Fe [43]. According to the relation

$$\lambda^s_{eff} = 1.5 (2\lambda_{100} + 3\lambda_{111})$$

(4)

results in 19 ppm, which is in excellent agreement with results on Fe$_{70}$Cr$_{30}$, keeping in mind that it is an HPT-deformed sample exhibiting a nanocrystalline microstructure. Furthermore, comparable results are found in the work of Bormio-Nunes et al. [8]. For the identical chemical composition of Fe$_{70}$Cr$_{30}$, they reported $\lambda_5$ values between 23 ppm and 47 ppm depending on the measured side of their cubic samples. The results of magnetostrictive measurements of annealed samples (500 °C for 1 h) are shown in Fig. 6. For all samples, the saturation magnetostriction increases compared to as-deformed samples. The signal for the Fe$_{50}$Cr$_{50}$ sample is slightly positive, the Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$ samples reach 12 ppm and 23 ppm, respectively. The increased magnetostriction with respect to the as-deformed state is thought to originate from a decreased number of crystal defects and a possible reduction of residual stresses [44] and further, analysis of EBSD scans (see Fig. 2) revealed a slightly stronger texture for the annealed sample, which could also enhance magnetostriction values [7,45,46]. Due to the large, positive values, a solid solution is still present for all FeCr compositions but no further improvement of $\lambda^s_{eff}$ by an increasing Cr content is found as motivated by Bormio-Nunes et al. [8]. During this work, we successfully produced nanocrystalline FeCr supersaturated solid solutions over a broad chemical composition and clarified that the alloys do not exhibit a two-phase structure in the nanocrystalline regime and confirmed the existence of a solid solution. Astonishingly a demixing into separated Fe and Cr phases was suppressed by a remarkable temperature stability of the solid solution up to 520 °C. As the principle aim was to decompose the microstructure into a nanocrystalline two-phase material, enabling the formation of an exchange biased material, a more complex annealing treatment and HPT-processing route has to be found, which is part of ongoing studies. Beforehand, adding substitutional elements could stabilise the microstructure even further and hinder grain growth. This could preserve the nanocrystalline structure during annealing, while a decomposition takes place.

### 4. Conclusion

Nanocrystalline FeCr alloys consisting of Fe$_{50}$Cr$_{70}$, Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$ are processed by HPT-deformation at RT. Upon annealing, the microstructure starts to coarsen between 300 °C and 500 °C, which is stronger pronounced for higher Fe contents. All materials investigated exhibit a hardening effect upon annealing with a peak between 300 °C and 400 °C followed by a softening at elevated annealing temperatures. In-situ HEXRD annealing experiments point towards a supersaturated solid solution of FeCr. However, concurrent finely dispersed Fe and Cr phases cannot be completely ruled out by microstructural investigations. Up to temperatures of 520 °C, no peak separation or broadening due to phase decomposition is found in HEXRD experiments. To clarify the ambiguous results from HEXRD experiments in regard of the prevailing phases, magnetostrictive measurements were conducted for HPT-deformed as well as subsequently annealed materials. Results show a clear deviation with respect to elemental Fe or Cr. The saturation
magnetostriiction $\lambda_s \mu_0 H = 2.257$ is 0 ppm (1 ppm), 8 ppm (12 ppm) and 17 ppm (23 ppm) for the as-deformed (500 °C annealed) alloys consisting of Fe$_{30}$Cr$_{70}$, Fe$_{50}$Cr$_{50}$ and Fe$_{70}$Cr$_{30}$, respectively. After excluding a magnetostriective contribution from the interface, these values are explained by the formation of a supersaturated solid solution by HPT-deformation, which persists during annealing and validates findings from the synchrotron HXRD measurements. To decompose these FeCr solid solutions and to enable exchange bias effects, more complex processing routes are necessary.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.jmmm.2021.168017.

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