On the effect of aluminum and chromium on the deformation twinning of body-centered cubic iron

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Abstract. This paper reports on investigations on the effect of aluminum and chromium on the formation of deformation twins in body-centered cubic iron. For this purpose, impact loading experiments (\( \dot{\varepsilon} = 10^4 \text{ s}^{-1} \)) were carried out at room temperature on samples of iron and the alloys Fe-10at.-% Al and Fe-10at.-% Cr. Furthermore, the grain size was varied in order to consider the effect of the alloying elements for both fine-grained and coarse-grained microstructures. The investigations on twin formation were carried out by electron backscatter diffraction (EBSD) and by quantitative image processing using optical microscopy. The observations show a comparable twin portion in iron and Fe 10at.-% Cr after impact loading for both grain sizes. In the alloy Fe-10at.-% Al the portion of twins is significantly increased for fine-grain and coarse-grain states. In order to clarify the possible reason, X-ray diffraction analyses (XRD) were additionally carried out. These show a shift in the reflexes which can be attributed to an elastic distortion of the lattice. This lattice distortion due to the formation of solid solution correlates very well with the determined twin fractions.

1 Introduction
Deformation twinning is an important mechanism of plastic deformation and is of great meaning for many metals in everyday technical life. In hexagonal closed packed (hdp) magnesium and magnesium alloys, the formation of twins is fundamental for the technical processability of the material [1]. In face-centered cubic (fcc), high-manganese TWIP steels (twinning Induced Plasticity), deformation twinning enables an extremely high ductility of 95% with tensile strength above 1.000 MPa [2–4]. This makes such steels of particular interest in components for automotive engineering.

Twinning can also be observed in body-centered cubic (bcc) iron materials. This is mainly the case at low temperatures below the room temperature and at impact loads with strain rates above \( \dot{\varepsilon} = 10^9 \text{ s}^{-1} \) [5,6]. For temperatures around room temperature and quasi-static loads, dislocation-based slip is the dominant deformation mechanism in bcc iron. The decreasing temperature or increasing strain rate leads to an increase in the shear stress required to activate twin formation or atomic slip. However, both effects have a clearly different dependence on temperature. While the required stress for dislocation-based slip increases with decreasing temperature, the stress for twin formation remains almost constant with decreasing temperature. The result is more hindered slip at low temperatures and thus the preferred deformation due to twin formation. From investigations of MEYERS ET AL., an overview of this correlation can be seen in Figure 1 [6]. Dislocation-based slip has no linear progression over temperature, so twinning is preferred below a critical temperature.
Figure 1. Critical stresses for the activation of dislocation-based slip and deformation twinning as a function of temperature and strain rate. The diagram shows a transition point from slip to twin formation below a critical temperature. The figure is based on calculations for iron with a grain size of 100 µm performed by MEYERS ET AL. [6].

Since NEUMANN’S discovery of deformation twins in iron meteorites in 1848, a large number of investigations on twin formation in metals have been carried out [6–11]. On the one hand, these studies focus on the effects of different influencing variables such as temperature and strain rate but also grain size. The grain size as an internal influencing variable is of particular importance. Several studies have already shown that an increase in grain size also has a favorable effect on twin formation. Similar to the temperature and strain rate, a grain size change also has a different effect on the preferred mechanism [12]. A schematic overview is shown in Figure 2. Therefore, the effect of grain size should also be considered in this study.

Figure 2. Schematic representation of the course of the formation stress for twin formation as well as displacement based sliding as a function of the grain size [12].
On the other hand, investigations on the nucleation mechanism of twinning are of great interest [13–22]. The theories available include both dislocation-based processes and mechanisms without dislocation involvement. The experimental proof of these formation mechanisms is currently in the focus of research. In addition to the use of experimental methods such as TEM and STEM, computer-based investigation methods are playing an increasingly important role in describing atomic motion processes during impact loading [23,24].

In the context of this study, investigations on the influence of alloying elements on the formation of deformation twins in bcc iron are presented. Specifically, the influence of aluminum and chromium on the formation of twins in bcc iron is investigated and compared with the twin formation in unalloyed iron. Similar to the influence of manganese in the already mentioned fcc TWIP steels, the effect of Al and Cr in the iron lattice will be investigated. Possible effects on twin formation are the influence on the stacking fault energy and an elastic lattice distortion. With respect to the portion of twins in the three materials, these effects will be considered and discussed.

2 Materials and methods
Iron (99.8% Fe) and iron alloys of Fe-10at.-% Al and Fe-10at.-% Cr are used for this study. In the following, the alloys subsequently abbreviated as Fe-10Al and Fe-10Cr. The experiments are carried out on the as-received state (cold-rolled) and on a heat-treated state. The respective grain size before and after heat treatment can be taken from Table 1. The grain size was determined using the line intersection method according to DIN EN ISO 643 and is compared with EBSD investigations. A more precise adjustment of the grain size was not possible for process-technical reasons, so that the scatter in the coarse grain sizes can be justified.

Table 1. Overview of grain sizes and heat treatment parameters for the three investigated materials Fe, Fe-10Al and Fe-10Cr

| Grain size         | Fe  | Fe-10Al | Fe-10Cr |
|--------------------|-----|---------|---------|
| fine-grained       | 44µm| 48µm    | 30µm    |
| coarse-grained     | 116µm (12 h @ 1150°C) | 140µm (1 h @ 950°C) | 201µm (10 h @ 1150°C) |

A mechanical test set-up is used to apply an impact load, which accelerates a hardened bolt to 50 m/s and applies a single load to the sample. The sample geometry is 6.0 mm x 6.0 mm (diameter x height). The bolt accelerates the samples, which are caught in a bullet trap 2.0 m away. The resulting plastic deformation is less than 1 % and can therefore be neglected for further considerations. A schematic representation of the experimental set-up shows Figure 3.

Figure 3. Schematic illustration of the experimental set-up of the set-up for impact loads.
In order to characterize the microstructure, metallographic preparations are required first. The metallographic preparation is carried out by hot mounting, grinding with SiC paper (up to grain size 2400) and polishing with diamond suspension (3 µm and 1 µm). To contrast the microstructure for subsequent optical microscopic examinations, a V2A-Beize is used for iron and Nital for the two iron alloys Fe-10Al and Fe-10Cr. For the EBSD analyses, the diamond polishing is not followed by contrasting, but by fine polishing with oxide particle suspension. Here, different suspensions are used depending on the material. An OP-S suspension is suitable for iron and Fe-10Cr, an OP-U suspension for Fe-10Al.

EBSD analyses are carried out for a random determination of the grain size as a comparison to the line section measurements as well as for the characterization of the crystallographic orientation conditions after the impact loading. With the help of EBSD, crystallographic properties can be determined which are used to reveal the preferred twin systems. This is possible by using the coincidence site lattice (CSL), which characterizes the crystallographic relationship between differently oriented areas. This method is particularly well suited for the study because of the misorientation between matrix and twin. The expected coincidence for the twin system in iron is the CSL-Σ3 [25].

Furthermore, possible plastic deformations before and after loading as well as second phases weren’t observed. The EBSD examinations were performed on a DualBeam FEI SCIOS with AMETEK-EDAX analysis. The selected scan areas have a size between 100 x 100 µm and 1000 x 1000 µm. The scan step size varies between 100 and 500 nm. In this work, the results are presented as color-coded figure of the inverse pole figure IPF.

EBSD measurements on the initial state show that the microstructure is single-phase and free of deformation. Deformation twins or a second phase are also not present. An overview of the crystallographic orientation conditions in the initial state shows Figure 4.

Figure 4. EBSD study of the three investigated materials Fe, Fe-10Al and Fe-10Cr in the initial state and the heat treated state.

The quantification of the twin portion (indicated as area fraction) is carried out using metallographic practices. Using optical microscopic images, the area fraction after impact loading are determined by image processing. The determination of the area fraction of twins is also possible using EBSD measurements. However, the section to be viewed is much smaller than in the optical microscopic section. For a representative and statistical evaluation, a high number of EBSD measurements per sample are necessary.

Binarized dark field images are used for quantification. These offer the advantage that grain boundaries and twin boundaries as well as microstructural artifacts and impurities are easier to detect. By software-based determination the portion of twins in several samples were calculated. This procedure is carried out for areas without and with deformation twins for each sample. The difference in detected signal (no microstructural feature = white, detected microstructural feature = black) between the two
parts of information (area fraction with twins $A_{\text{twins}}$ and area fraction without twins $A_{\text{no twins}}$) shown in Figure 5 automatically results in the pure portion of deformation twins.

![Figure 5. Procedure of quantitative measurement of twinning portion $A_{\text{twins}}$ (left) und $A_{\text{no twins}}$ (right) using binarized dark field images.](image)

X-Ray Diffraction (XRD) analyses are used to determine the structure and phase characteristics of the produced alloys Fe-10Al and Fe-10Cr. Thus it can be ensured that the investigated materials are present in a single-phase, body-centered cubic lattice.

Furthermore, information about the influence of the alloying elements on the iron lattice can be derived from the XRD results. This results are discussed in the results section in connection with the optical microscopic analyses. The sample preparation of the XRD measurements is analogous to the metallographic preparation up to the deformation-free polished state. The used radiation for the XRD analysis is Co-Kα1. The diffraction diagrams show a normalized representation of the intensities.

3 Results and Discussion

The results of the impact loaded materials (Fe, Fe-10Al and Fe-10Cr) shown below, reveal the formation of deformation twins in the microstructure in all three materials and both grain sizes. First of all, the EBSD investigations show that the formed twins have an orientation difference to the initial crystal (matrix) of 59° and are thus a twin boundary of the type CSL-$\Sigma 3$. From this it can be deduced that $<1\overline{1}1> \{1\overline{1}2\}$ is the preferred twin systems in all three materials. There is no change in the twin system by alloying Al and Cr. As an example, Figure 6 shows EBSD overview measurements of the three materials with fine-grained and coarse-grained microstructures. The random distribution of twins can be seen. A preferred crystallographic orientation in which twinning takes place is not observed.

![Figure 6. EBSD study of the three investigated materials Fe, Fe-10Al and Fe-10Cr with fine-grained and coarse-grained microstructure after impact loading.](image)
Table 2 presents the results of the measured twin amount after impact loading. The determined portion of twins for iron with fine-grained microstructure is 3.1 %. For the alloy Fe-10Cr the portion is 3.4 % on average and thus in a similar range. In comparison, the determined portion of twins in the material Fe-10Al is 7.9 % on average. For the coarse-grained microstructure, the investigations show an average portion in iron of 6.5 % and for Fe-10Cr of 7.5 %. For Fe-10Al the average portion is 9.1 %. For both grain size it can be observed that the alloy Fe-10Al has a higher portion of twins. For Fe-10Cr a preferred effect on the formation of twins can also be observed, but this is much less pronounced.

In addition, there is the superimposition of the grain size effect. As already mentioned, an increasing grain size leads to preferred twinning. This effect can also be observed in the comparison of fine and coarse-grained microstructure. In the fine-grained state, the average grain sizes determined are 44 µm for iron, 48 µm for Fe-10Al and 30 µm for Fe-10Cr. The differences in grain size are small and therefore exert little influence on the formation of twins. A different situation exists for the coarse-grained microstructure due to the deviation in grain sizes. The grain sizes of iron (116 µm), Fe-10Al (140 µm) and Fe-10Cr (201 µm) have a significantly higher absolute deviation. It is therefore assumed that with similar grain sizes, e.g. around 200 µm, iron and Fe-10Cr would be closer together (~ 7.0 %) and the twin portion in Fe-10Al would increase to ~10.0 % or higher.

Table 2. Overview of the twin portion with standard deviation in Fe, Fe-10Al and Fe-10Cr after impact loading depending on the grain size

| Grain size        | Fe     | Fe-10Al | Fe-10Cr |
|-------------------|--------|---------|---------|
| fine-grained      | 3.1 ± 0.8 | 7.9 ± 1.0 | 3.4 ± 1.0 |
| coarse-grained    | 6.5 ± 2.6 | 9.1 ± 0.9 | 7.5 ± 2.7 |

The XRD investigation were done on the initial material with the fine-grained microstructure. A section of the obtained diffraction diagram is shown in Figure 7. The overlaid diffraction diagram shows the (011) reflexes for Fe, Fe-10Al and Fe-10Cr in the angular range 50°...2θ...55°. The measurements reveal the influence of the alloying elements in the iron lattice. A shift in the position of the reflexes are detected, which can be explained by the elastic distortion of the lattice. The lattice parameter of the (011) reflex in iron is 202.68 nm. With 10 at.-% Cr this shifts to 203.05 nm, for 10 at.-% Al to 203.85 nm. The increase of the lattice parameter correlates very well with the determined twin fractions in both the fine-grained and coarse-grained state. The XRD investigations thus provide a good explanation for the observed twin formation behavior.
Figure 7. Diffraction diagram from XRD investigations on the three materials Fe, Fe-10Al and Fe-10Cr in the unloaded initial state. The normalized intensity for the (011) reflex in the angular range 50°...2θ...55° is shown. It reveals a peak shift resulting from an elastic lattice distortion due to the addition of Al and Cr.

The XRD measurements confirm an influence of the alloying elements Al and Cr on the iron lattice and provide an explanation for the observed twin formation. Which effect is caused by the alloying elements on the stacking fault energy cannot be fully answered by this study.

On the one hand, there are methodological challenges in such nanoscopic scale investigations. The direct determination of the stacking fault energy by means of TEM investigations as well as the indirect determination from XRD measurements should be the aim in future studies.

Furthermore, the question of the existence of stacking faults in bcc iron is not entirely solved. VITEK was one of the first to deal with the existence of stable stacking faults in the bcc lattice [26,27]. The work suggests that stable intrinsic stacking faults in the bcc lattice are not likely. Even the formation of a single-layer stacking fault would not bring any energetic advantages. This fact also includes the preferred (112) plane for twin formation. However, extrinsic stacking faults are not excluded, which are formed as a result of dislocation dissociation and are stable. A generalized stacking fault energy (GSF energy) has therefore been defined to describe dislocation dissociation. This approach has been developed in a large number of computer-aided investigations using various methods [28]. In principle, the question of the existence of stacking faults in bcc iron is not completely clarified and requires a large number of further investigations, also with regard to their influence on the formation of twins in bcc iron.

4 Summary
In this study the influence of Al and Cr on the formation of twins in bcc iron is reported. Samples of the three materials Fe, Fe-10 at.% Al and Fe-10 at.% Cr were impact loaded at a strain rate of $10^4$ s$^{-1}$ at room temperature. Furthermore two different grain sizes were considered instead of. Fine-grained in the range from 30 µm to 48 µm and coarse-grained in the range from 116 µm to 201 µm. The investigations by EBSD first show that in all materials and grain size states the misorientation between twin and crystal matrix is ~59°. It can be concluded from this that the preferred twin system is the $<111>$ {112}. The optical microscopic analyses show that for both states, Fe-10Al has the largest portion of twins after impact loading. For fine-grained state the mean portion is 7.9 % and for the coarse-grained 9.1 %. For the other two materials, the percentages are lower. For fine-grained microstructure, the mean portion is
comparable and is 3.1 % for Fe and 3.4 % for Fe-10Cr. In the coarse-grained microstructure, the mean portion are 6.5 % for Fe and 7.5 % for Fe-10Cr. However, it can be assumed that the difference in the coarse-grained state is an influence which must be taken into account. Suppose, that the grain sizes are identical (deviation below 10 µm), it can therefore be assumed that Fe and Fe-10Cr have a tendency to form similar twin portions and Fe-10Al has a much stronger tendency to form twins. XRD investigations show a shift of the peak positions in the diffraction diagram. An example is the shown reflex (011). The increase of the lattice parameter from 202.68 nm (Fe) to 203.05 nm (Fe-10Cr) or to 203.85 nm (Fe-10Al) is in very good agreement with the shown twin portions. For a validation of the effect, further experiments with identical grain sizes are useful. However, basic manufacturing and process engineering approaches must be revised in the preliminary field.

Furthermore, an expansion of the alloying elements and the alloy contents is considered useful in order to investigate the effect for other alloys. In principle, the experimental investigation of possible stacking fault formations by TEM and XRD studies is also considered useful for future studies. In addition, molecular dynamic simulations could provide further information about the effect of alloy atoms on twin nucleation and twin growth.

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