FINAL REPORT

1 General Information

DFG reference number: 655603

Project number: GR 4174/5-1

Project title:
Structure-properties relations in single phase fcc and bcc high entropy alloys under a tribological load

Name(s) of the applicant(s): Prof. Dr. rer. nat Christian Greiner

Official address(es):

Prof. Dr. rer. nat Christian Greiner
Straße am Forum 7; Geb. 30.49
76131 Karlsruhe

Name(s) of the co-applicants:

Prof. Dr.-Ing. Martin Heilmaier
Engelbert-Arnold-Straße 4; Geb. 10.91
76131 Karlsruhe

Name(s) of the cooperation partners:
Dr. Stefan Eder, Prof. Christian Kübel, Dr. Michael Feuerbacher, Dr. Alexander Kauffmann, Alexander Dyck, Prof. Clelia Righi

Reporting period (entire funding period): 19.12.2018 – 31.12.2023

2 Summary

Tribological contacts describe two bodies in relative motion to each other. Such contacts occur basically everywhere in our everyday life, between shoes and floors, in joints and also in technical application e.g. in all kind of machinery. 23 % of the total energy is used to either overcome unwanted friction or to replace worn parts. This number has to be reduced. In this work, the aim is to understand fundamentally the microstructural evolution in the sub-surface area of metals and alloys under tribological loading as microstructural evolution can influence the friction coefficient as well as the wear particle formation. The end-goal is to be able to adjust these processes, in order to on the long run allow for tailored tribological behaviour.
Shear stress-controlled deformation mechanisms determine the microstructural evolution. Therefore, it has to be known which crystallographic systems are activated. In material science, the activated crystallographic system is determined by calculating the resolved shear stress on each system and the one(s) with the highest resolved shear stresses are activated. The same should be done for tribological loading, but a reliable stress field model is missing to calculate the resolved shear stresses. The work performed through this grant added probes to the deformation layer to test various stress field models. As probes deformation twins has been used as these have two critical differences from dislocation motion: 1) twins are 3D extended defects with a distinct strain release; 2) twin formation is dependent on the crystallographic orientation, therefore exhibiting a pronounced tension-compression anisotropy. After some pre-tests on polycrystalline CoCrFeMnNi, experiments on single crystalline CoCrFeMnNi with careful chosen initial crystal orientation have been conducted. Different counter bodies have been used to change the friction coefficient.
By doing so, data sets were created which are excellent to test various stress field models. The stress field models can be grouped into 1) single stress components, 2) analytical linear elastic model (Hamilton) 3) FE-simulations with varying material models. For the experiments resulting in high friction coefficients, the normal stress in sliding direction as well as the shear stress in sliding direction was identified to be the determining stress components for twin activation. The Hamilton stress field predicted twin formation on the experimentally identified twin systems with considering crystal rotation. For a low friction coefficient, the FE-simulations considering plasticity are required. Even as these model predictions are close to the experimental results, they do not fully agree. The results obtained through this grant are the very first to systematically investigate the stress field models. Even more importantly, the first experimental probe was developed that allows to systematically and unambiguous put stress field models for tribological loading to the test. This new capability will prove invaluable for the future development of materials tribology.

Tribologie beschreibt die Wissenschaft von zwei Körpern in Relativbewegung zueinander. Solche Kontakte treten im Grunde überall in unserem Alltag auf: zwischen Schuh und Boden, in Gelenken und auch in technischen Anwendungen. 23 % der gesamten Energie muss aufgewandt werden um unerwünschte Reibung zu überwinden oder abgenutzte Teile zu ersetzen. Diese Zahl gilt es zu reduzieren. In dieser Arbeit ist es das Ziel, die mikrostrukturelle Entwicklung im oberflächennahen Bereich von Metallen und Legierungen unter tribologischer Belastung grundlegend zu verstehen, da diese den Reibungskoeffizienten sowie die Bildung von Verschleißpartikeln beeinflussen kann. Das Endziel ist es, diese Prozesse so anzupassen, dass auf lange Sicht ein maßgeschneidertes tribologisches Verhalten ermöglicht wird.
Scherspannungsaktivierte Deformationsmechanismen bestimmen die mikrostrukturelle Entwicklung. In der Materialwissenschaft wird das aktivierte kristallographische System bestimmt, indem die auf jedes System projizierte Scherspannung berechnet wird, und diejenigen mit den höchsten projizierten Scherspannungen werden aktiviert. Derselbe Ansatz ist für die tribologische Belastung angedacht, allerdings fehlt es an einem Spannungsfeldmodell, um die projizierten Scherspannungen zu berechnen. Dieses Projekt verwendet deshalb Marker in der De-

formationsschicht, um verschiedene Spannungsfeldmodelle zu testen. Als Marker wurden Deformationszwillinge verwendet, da diese zwei wesentliche Unterschiede zur Versetzungsbewegung aufweisen: 1) Zwillinge sind dreidimensionale Defekte mit einer unidirektionalen Dehnungsfreisetzung; 2) die Zwillingsbildung ist von der kristallographischen Orientierung abhängig und zeigt daher eine ausgeprägte Zug-Druck-Anisotropie. Nach Vorversuchen an polykristallinem CoCrFeMnNi wurden Experimente an einkristallinem CoCrFeMnNi mit sorgfältig gewählten anfänglichen Kristallorientierungen durchgeführt. Verschiedene Gegenkörper wurden verwendet, um den Reibungskoeffizienten zu verändern.
Auf diese Weise wurde ein Datensatz erstellt, der sich hervorragend eignet, um verschiedene Spannungsfeldmodelle zu testen. Die Spannungsfeldmodelle können in 1) einzelne Spannungskomponenten, 2) analytisches lineares elastisches Modell (Hamilton) und 3) FE-Simulationen mit variierenden Materialmodellen gruppiert werden. Für die Experimente, die zu hohen Reibungskoeffizienten führten, wurde die Normalspannung in Gleitrichtung sowie die Scherspannung in Gleitrichtung als bestimmende Spannungskomponenten für die Zwillingsaktivierung identifiziert. Das Hamilton-Spannungsfeld sagte die Zwillingsbildung auf den experimentell identifizierten Zwillingssystemen unter Berücksichtigung der Kristallrotation voraus. Für einen niedrigen Reibungskoeffizienten sind die FE-Simulationen unter Berücksichtigung der Plastizität erforderlich. Auch wenn diese Modellvorhersagen nahe an den experimentellen Ergebnissen liegen, stimmen sie nicht vollständig überein. Die durch diese Arbeit erzielten Ergebnisse sind die ersten, die systematisch die Spannungsfeldmodelle untersuchen. Noch wichtiger ist, dass der erste experimentelle Marker entwickelt wurde, die es ermöglicht, Spannungsfeldmodelle für tribologische Belastungen systematisch und eindeutig zu testen. Diese neue Fähigkeit wird sich als unschätzbar wertvoll für die zukünftige Entwicklung der Materialtribologie erweisen.

3 Progress Report

The aim of the original proposal was to investigate the tribological behaviour of the material class named High Entropy Alloys (HEA), based on their promising properties. Therefore, the best investigated fcc and bcc HEA were suggested, which are CoCrFeMnNi and HfNbTaTiZr, respectively. All the sample materials were manufactured in the labs of Prof. Dr. Heilmaier. In the following not all the conducted experiments are mentioned such as varying the sliding velocity, experiments in liquid nitrogen and at different rolling degrees. Throughout the whole funding period Prof. Dr. Heilmaier and Dr. Alexander Kauffmann were close collaborators. During the investigation of CoCrFeMnNi, it turned out that this is a perfect model material to generate fundamental knowledge about the stress field acting under a tribological load. It turned out that being able to probe and understand a wide variety of possible stress field models would have infinitely more impact on the materials tribology community than following the initial plan to determining the tribological properties of two alloys. We therefore made the deliberate and strategic decision to deviate from the original research plan.

CoCrFeMnNi is a perfect model material for the purpose of developing an experimental probe for tribological stress field models as it i) is fcc ii) has a medium stacking fault energy and iii) is highly sensitive towards the tribological system. Herby, the fcc crystal structure is of advantage as the PI's group has a high expertise in the field of the microstructural evolution in fcc metals. The medium stacking fault energy results in the formation of deformation twins at room temperature with moderate strain rates. These play a pivotal role in this research, as they are used as probes within the deformation layers for the stress field. The advantage of the sensitivity towards the tribological system is that a large variety of friction coefficients can be achieved as can be seen in Figure 1, which is difficult to achieve for other alloy systems. The first experiments were conducted on polycrystalline CoCrFeMnNi to get a ballpark figure for the tribological properties. All experiments where conducted in a single trace fashion with a sliding speed of 0.5 mm/s. The remaining experimental parameters are presented in the legend of Figure 1.

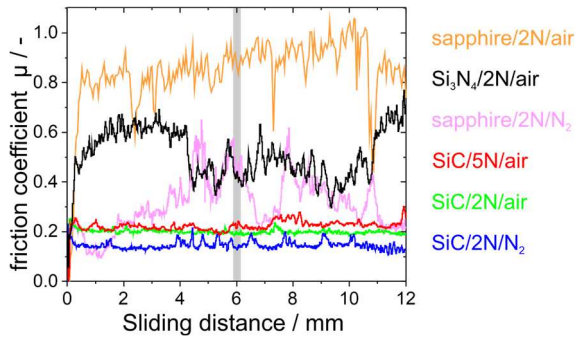

Figure 1. Friction coefficient over the sliding distance. The different colours refer to various experimental set-ups concerning, counter body material, atmosphere and normal load. The grey rectangle marks the TEM foil lift-out position.

Here, different counter body materials and a change in ambient atmosphere have influenced the friction coefficient, whereby the change of normal load has no influence on the friction coefficient. With high friction coefficients material transfer on the counter body was observed, being a clear indicator for high adhesive forces. The exact reasons for the measured friction coefficients for the specific material pairings remain unclear, possible influencing parameters are surface roughness, surface energy and surface softening based on the variation in humidity. Although, the reasons of low and high friction are unclear, the aim of altering the stress field is achieved with this set-up; meaning that the influence of the friction coefficient on the microstructural evolution can be studied. TEM foils parallel to the sliding position and in the middle of the wear track (position marked by the grey rectangle within Figure 1) were cut und STEM images taken. These are presented in Figure 2. For some experimental set-ups, microstructures of two initial grains are shown by bright and dark colours in Figure 2. TKD measurements were conducted to analyse the original crystal orientation, the crystal rotation and the newly formed grains. HR-TEM, conducted by the group of Prof. Dr. Christian Kübel at KIT's KNMFi (made possible through a KIT internal proposal), was required to analyse the lines tilted in sliding direction in Figure 2b, which were detected as twins [1]. This data was used to understand the influence of crystal orientation, normal load and friction coefficient. The initial grain orientation can strongly influence the deformation mechanism, especially in the case of deformation twinning which was known in the literature for uniaxial loading [2–5]. The influence of the initial grain orientation under tribological load was investigated by comparing two subsurface deformation layers with the same tribological system and therefore, also the same applied stress field. The STEM images in Figure 2a and b are ideal for the described scenario. The line-type features tilted in sliding direction were identified as twins. This means that while one grain exhibits twins and the other does not. Therefore, also a grain orientation dependency on the formation of twins exists for tribological loading. However, at this point in our research, the relations between crystal orientation and twin formation was unknown.

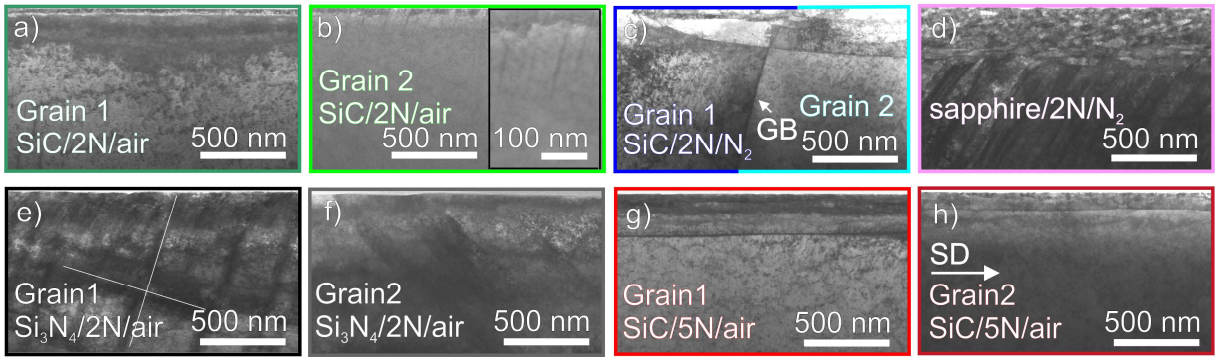

Figure 2. STEM images of deformation layers after single trace experiments with the variation of counter body material, atmosphere, normal load and crystal orientation. The sliding direction is from left to right.

Concerning the normal load, the experiments SiC/2N/air and SiC/2N/N_2 can be compared to SiC/5N/air as the influence of the atmosphere is marginal with SiC as counter body material. By comparing these experiments, the deformation depth appears to be more dependent on

the crystal orientation than on the normal load. However, higher misorientations are measured at the formed grain boundaries, being a sign for higher dislocation activity in the surface near region with a higher normal load. The increase in friction coefficient demonstrates a significant impact on the deformation layer, as the comparison of the microstructural characteristics caused by SiC and sapphire as counter body shows. Based on analyses of the crystal orientation, crystal rotation and the occurrence of twins, the following hypotheses were proposed: i) for low friction coefficients: twins are caused by the compressive normal stress parallel to the normal direction and ii) for high friction coefficients: twins are caused by the tensile normal stress parallel to the sliding direction.

These hypotheses needed to be systematically investigated. Therefore, single crystalline CoCrFeMnNi samples, provided by Dr. Michael Feuerbacher, were employed. The tensile-compression anisotropy of deformation twins was utilized to test the proposed hypotheses allowing insight into the stress field under tribological loading. Crystal directions which form twins under unidirectional load under tension or compression were systematically paired. These deliberations resulted in chosen the following crystal orientations: ND $[00\bar{1}]$ SD $[100]$, ND $[00\bar{1}]$ SD $[\bar{1}10]$, ND $[0\bar{1}\bar{1}]$ SD $[100]$, ND $[0\bar{1}\bar{1}]$ SD $[0\bar{1}1]$ and ND $[0\bar{1}\bar{1}]$ SD $[\bar{2}\bar{1}1]$. To alter the stress field, SiC and sapphire balls were both used for single trace experiments with a constant normal load of 2 N and a sliding speed of 0.5 mm/s in air atmosphere with 50 % RH. The resulting friction coefficients are presented in Figure 3. The friction coefficients with SiC as counter body is low with 0.2 and show little deviation with the crystal orientation. In sharp contrast, with sapphire as counter body, the friction coefficients are much higher and varies with the crystal orientation. This somewhat surprising difference needed to be investigated and understood. As we assume that minute differences in surface composition and chemistry are the root causes, a collaboration with Prof. Clelia Righi from the University of Bologna was established. Prof. Righi is an expert in ab initio and density functional theory based simulations. We are certain that this international collaboration will be very fruitful and an exciting long term result of this grant.

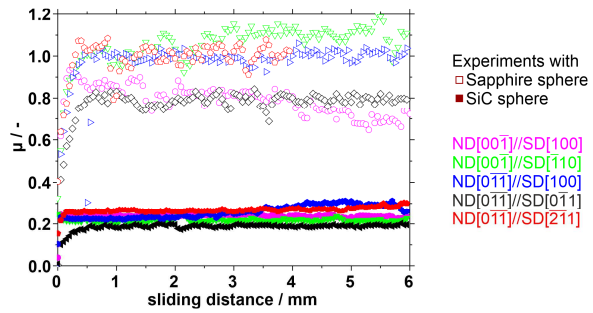

Figure 3. Friction coefficient μ as a function of the sliding distance with single crystalline CoCrFeMnNi. The different crystal orientations are marked by the colour coding and the used counter body by hollow and full symbols.

Experimentally the main goal was achieved by this approach: Altering the stress field by varying the counter body material. This allows to investigate the influence of the friction coefficient on the microstructural evolution. The resulting subsurface deformation layers investigated parallel to the sliding direction are presented in Figure 4. The difference in friction coefficient strongly influences the deformation layer depth and its characteristics. The maximum deformation layer thickness with a friction coefficient of 0.2 is less than 0.6 µm, while a deformation depth up to 20 µm is reached with a friction coefficient of ~ 1. The microstructural characteristics differ depending on the friction coefficient and the crystal orientation: With a low friction coefficient, lines parallel to the surface were observed for all crystal orientations. These were analysed as either small or large angle grain boundaries via transmission Kikuchi diffraction and are referred to as a so-called dislocation trace line [6]. Additional lines tilted in sliding direction were observed for the crystal orientations ND $[00\bar{1}]$ SD $[\bar{1}10]$ and ND $[0\bar{1}\bar{1}]$ SD $[100]$. HR-TEM was required to analyse them. The FFTs of the HR-TEM show that these lines are rich in stacking faults or twins. As all mechanisms for twin formation are based on stacking faults, the lines are most likely twins or a precursor of them. In contrast, the deformation layers caused with sapphire spheres (high friction coefficient) is much more pronounced. TKD measurements have revealed nanocrystalline grains with large angle grain boundaries right below the surface. With increasing distance from the surface

in sliding direction, elongated subgrains with low angle grain boundaries are observed for many crystal orientations. With a further increase in distance to the surface, the TKD measurements of all crystal orientations show a colour gradient based on crystal rotation. In four of five crystal orientations, lines tilted in sliding direction are observed. These lines were detected as deformation twins as shown by the blue coloured grain boundaries. Deformation twins were formed for the low friction coefficient with these crystal orientations: ND $[00\bar{1}]$ SD $[\bar{1}10]$ and ND $[0\bar{1}\bar{1}]$ SD $[100]$, and with a high friction coefficient for these crystal orientations: ND $[00\bar{1}]$ SD $[\bar{1}10]$, ND $[0\bar{1}\bar{1}]$ SD $[100]$, ND $[0\bar{1}\bar{1}]$ SD $[0\bar{1}1]$ and ND $[0\bar{1}\bar{1}]$ SD $[\bar{2}\bar{1}1]$. The activated twin system for each crystal orientation was additionally analysed.

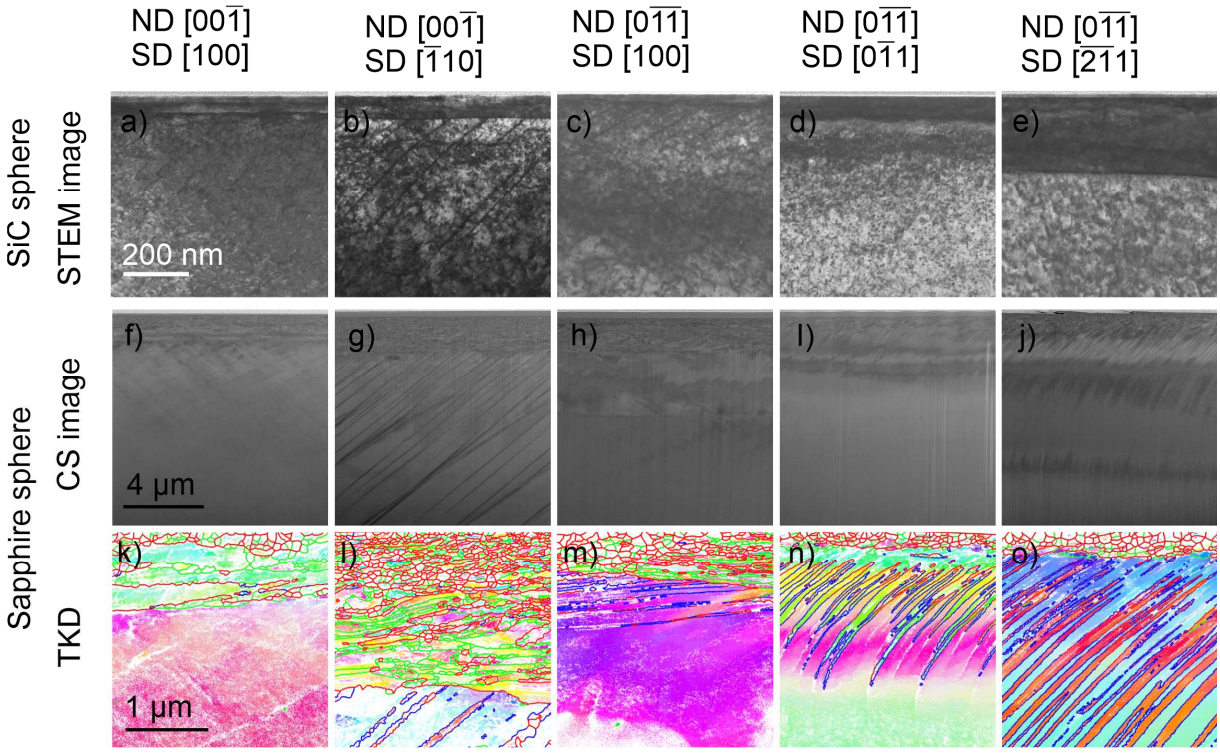

Figure 4. Deformation layers depending on the friction coefficient and the crystal orientation cut in the middle of the wear track parallel to the sliding direction. Each column shows the results of one crystal orientation. a)-e) STEM images of the deformation layers with SiC sphere as counter body (= low friction coefficient), f)-j) cross-section images of the deformation layers with sapphire sphere as counter body (= high friction coefficient), k)-o) TKD measurements of the deformation layers with sapphire sphere as counter body, green lines are small angle grain boundaries, red lines are large angle grain boundaries and blue lines are twin boundaries. The sliding direction is from left to right. The scale bar is given in the first image of each row.

The hypothesis for the low friction coefficient is that the normal stress parallel to the normal direction causes twin formation. If this were true, crystal orientations with ND $[00\bar{1}]$ would exhibit twins, whereas those with ND $[0\bar{1}\bar{1}]$ not. The experimental results clearly do not support this hypothesis! For a high friction coefficient, a tensile normal stress parallel to the sliding direction was proposed to lead to twin formation. Therefore, twins are expected for crystal orientations with SD $[\bar{1}10]$, SD $[0\bar{1}1]$ and SD $[\bar{2}\bar{1}1]$. Again, this is not supported by the experimental results! This unambiguously demonstrates the necessity of a 3D stress field model to understand the stresses acting under tribological loading. The first 3D stress field model that comes to mind is the one published by Hamilton [7]. He superimposed the Hertzian stress field with a shear stress proportional to the friction coefficient measured within the experiments. A limitation of the described stress field is the linear elastic material model. Nevertheless, this approach has been widely used in the literature and also employed by us to calculate the resolved shear stresses on all the twelve twin systems of the fcc lattice. The twin systems were defined such that a positive resolved shear stress leads to twin formation, because a negative

resolved shear stress would result in AA stacking, which is energetically not possible. The maximum resolved shear for each crystal direction in depending on the twin system is given in Figure 5 for the five crystal orientations and both friction coefficients. First, the differences in the resolved shear stress values are twice as high with the higher friction coefficient. The friction coefficient influences the distribution of the resolved shear stress for the five crystal orientations. ND $[00\bar{1}]$ SD $[\bar{1}10]$ and ND $[0\bar{1}\bar{1}]$ SD $[100]$ (green and the blue coloured crystal orientation) exhibit twins with the low friction coefficient and therefore these two bars have to be the highest, if the Hamilton stress field model was valid. This is not the case. Therefore, the Hamilton stress field cannot be used for the prediction of twin formation with low friction coefficients and therefore does not successfully represent the stresses acting under the moving sphere. As the Hamilton stress field is the by far most employed approach in the literature and materials tribology community, this result of ours clearly demonstrated the need for a new methodology. We therefore reached out to PD Dr. Stefan Eder from TU Vienna, who is an expert in the molecular dynamics modelling of tribological contacts. MD simulations performed by Dr. Eder allowed a fresh interpretation of the Hamilton stress field. If one takes into account the resolved shear stress distribution calculated with the Hamilton stress field on the experimentally identified twin system at the position relative to the moving sphere where the MD simulations predict the twin formation to happen, there then is excellent agreement between model and experimental result [8]. This is an exciting and very helpful result as it for the first time allows to have agreement between stress field model and experiment. It also indicates that the position of twin formation in reference to the ball and the stress components activating twins are dependent on the crystal orientation.

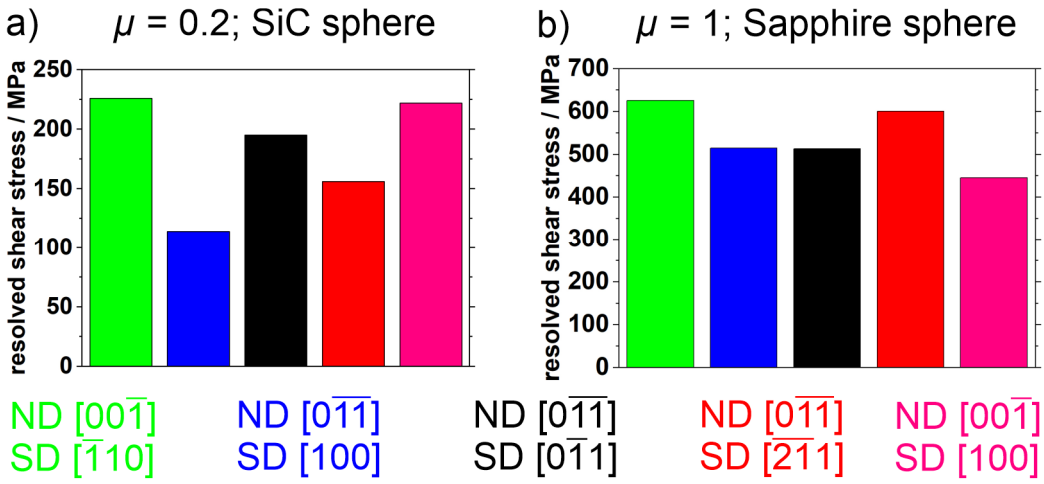

Figure 5. Calculated resolved shear stresses with the stress field model for tribological loading published by Hamilton; the calculations were conducted with the experimentally measured friction coefficients, namely a) μ = 0.2, b) μ = 1. The colour coding indicated the investigated crystal orientations. For each crystal orientation, the maximum resolved shear stress on an arbitrary twin system is given.

For the experiments with the high friction coefficient, the only crystal direction without twins is ND $[00\bar{1}]$ SD $[100]$. This crystal orientation has also the lowest resolved shear stress in Figure 5b. The crystal orientation data of the experiment with a sapphire ball with counter body show a large crystal rotation within the TKD measurements. For this reason, the twin systems with the maximum resolved shear stress were determined with the initial crystal orientation and with the final rotated crystal orientation. The experimentally determined twin plane and the twin systems with the highest resolved shear stress are given in Table 1. The initial and the rotated crystal orientation lead to the same twin system with the highest resolved shear stress for ND $[00\bar{1}]$ SD $[\bar{1}10]$ and ND $[0\bar{1}\bar{1}]$ SD $[100]$. The twin systems fit to the experimentally identified plane. With ND $[0\bar{1}\bar{1}]$ SD $[0\bar{1}1]$, four twin systems exhibit the same resolved shear stress with

the initial crystal orientation. The crystal rotation result into the selection of one twin system, which corresponds to the experimentally identified twin plane. For ND $[0\bar{1}\bar{1}]$ SD $[\bar{2}\bar{1}1]$, the experimentally identified twin plane neither fit to the twin system exhibiting the highest resolved shear stress with the initial crystal orientation nor with the rotated one.

Table 1. For the crystal orientations exhibiting twins, the experimentally identified twin plane and the twin systems with the highest resolved shear stresses using the initial crystal orientation and the rotated crystal orientation (extracted from experimental data).

	Twin plane Experiment	**Initial crystal orientaion**	**rotated crystal orientation**
ND$[00\bar{1}]$//SD$[\bar{1}10]$	$(1\bar{1}\bar{1})$	$(\bar{1}11)[\bar{1}1\bar{2}]$	$(\bar{1}11)[\bar{1}1\bar{2}]$
ND$[0\bar{1}\bar{1}]$//SD$[100]$	(111)	$(\bar{1}\bar{1}\bar{1})[2\bar{1}\bar{1}]$	$(\bar{1}\bar{1}\bar{1})[2\bar{1}\bar{1}]$
ND$[0\bar{1}\bar{1}]$//SD$[0\bar{1}1]$	$(11\bar{1})$	$(\bar{1}11)[\bar{1}1\bar{2}]$ $(\bar{1}1\bar{1})[\bar{1}12]$ $(\bar{1}\bar{1}\bar{1})[\bar{1}\bar{1}2]$ $(11\bar{1})[112]$	$(11\bar{1})[112]$
ND$[0\bar{1}\bar{1}]$//SD$[\bar{2}\bar{1}1]$	$(\bar{1}\bar{1}1)$	$(\bar{1}\bar{1}\bar{1})[2\bar{1}\bar{1}]$	$(\bar{1}11)[211]$

For this reason, the crystal rotation was further investigated for ND $[0\bar{1}\bar{1}]$ SD $[\bar{2}\bar{1}1]$. In Figure 6, the resolved shear stress on all twin systems are presented as a function of the rotation angle around the TD. 40° is hereby the maximum rotation a ngle from the experimental data. At 0° the twin system $(\bar{1}\bar{1}\bar{1})[2\bar{1}\bar{1}]$ has the highest resolved shear stress and at 40° $(\bar{1}11)[211]$ as given in Table 1. In between, another twin system has the highest resolved shear stress, namely $(11\bar{1})[112]$. The plane of the mentioned twin system fits to the experimentally identified one. These analyses show the influence of crystal rotation, which is not intrinsically included in the Hamilton stress field. As crystal rotation changes the position of slip and twin systems, the activated systems may be altered during rotation. Therefore, a model for crystal rotation describing the rotation axis and the angle as a function of the crystal orientation is required in the future; a new research avenue identified as a result of this grant.

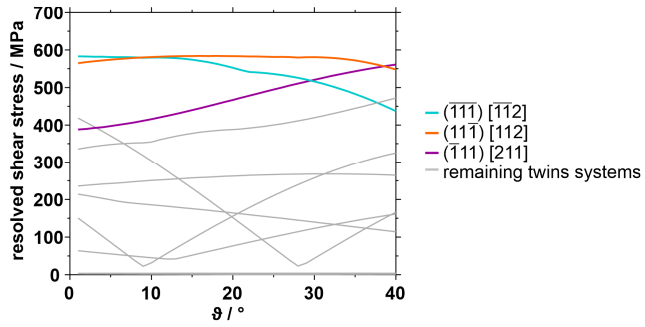

Figure 6. Resolved shear stresses on the twin system for the crystal orientation ND $[0\bar{1}\bar{1}]$ SD $[\bar{2}\bar{1}1]$ as a function of ϑ. ϑ is herby the rotation angle around TD.

The normal stress parallel to the sliding direction and shear stress in sliding direction were identified as the stress components having the most pronounced influence on twin formation. In all the reasoning above, literature values for the critical resolved shear stress for twin formation, which is 110 MPa up to 378 MPa [9–12], have not been considered with the high friction coefficient. In Figure 5b, higher resolved shear stresses were calculated for all crystal orientations including ND $[00\bar{1}]$ SD $[100]$ without any observed twins. As the Hamilton stress field is at its heart a linear elastic stress field, an overestimation of the resolved shear stresses is inevitable. Summing up the evaluation of the usefulness of the Hamilton stress field, we can undoubtedly state that it has its limitations. If one combines it with MD simulations however, it allows for useful predictions. In exactly herein lies the catch: It is not feasible to contact an MD simulation – which would require accurate potentials and potentially is computationally expensive – for each contact scenario. We therefore aimed at testing an approach with a much lower threshold. This was identified in finite element modelling, which could also be combined with crystal plasticity approaches if needed. To translate these thoughts into reality, we reached to Prof. Thomas Böh-

lke from KIT's institute for engineering mechanics. These FE-based simulations were only conducted for the low friction coefficient scenarios. In the literature (CP)-FEM analyses have already been used to simulate the tribological stress field. All these studies however only used one material model and compared the results to experimental data with limited success [13–15]. The unique aspect of our approach is the systematic variation of the material model. The yield stress, the work hardening behaviour as well as the elastic and plastic anisotropy have all been systematically varied and investigated. The employed material parameters are presented in Table 2.

Table 2. Materials parameters used for the (CP-) FEM simulations.

Designation	**HYLW**	**LYHW**	**LYLW**	**LYnoW**	**CP**
Elastic model	isotropic	isotropic	isotropic	isotropic	anisotropic
	E = 203 GPa [16] ν = 0.25 [16]				$C_{\{11\}}$ = 196 GPa [17] $C_{\{12\}}$ = 118 GPa $C_{\{44\}}$ = 130 GPa
Plastic model	isotropic	isotropic	isotropic	isotropic	anisotropic
Yield strength	high	low	low	low	Low
	R_{es} = 360 MPa [18]	R_{es} = 125 MPa [18]			τ_{sl} = 43 MPa [11]
Work hardening	low [16]	high [16]	low [16]	none	none

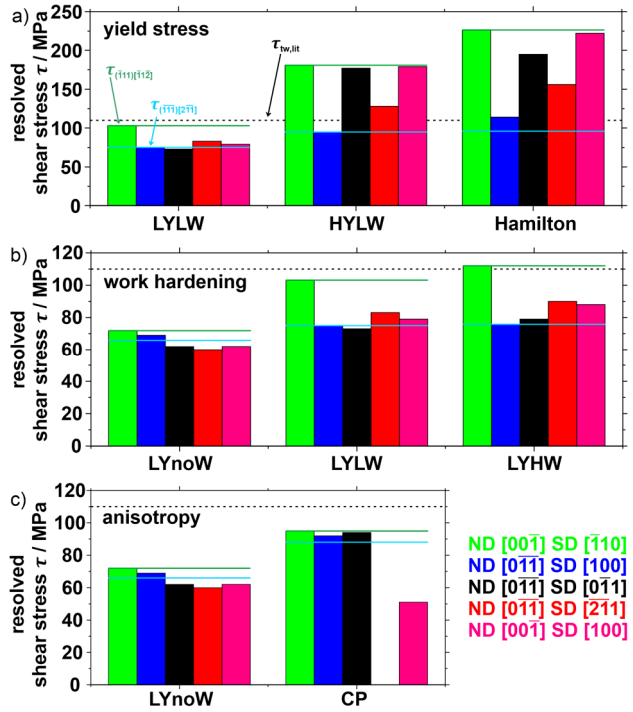

Figure 7. Resolved shear stress in dependency of the used crystal orientation and systematically varied material models. a) influence of the yield stress, b) influence of work hardening and c) influence of anisotropy. The dashed line is the critical resolved shear stress for twin activation from literature $\tau_{tw,lit}$. The experimentally identified twin system for the crystal orientation ND $[00\bar{1}]$ SD $[\bar{1}10]$ is $(\bar{1}11)[11\bar{2}]$. $(\overline{111})[2\bar{1}1]$ is the experimentally identified twin system for the crystal orientation ND $[0\bar{1}1]$ SD $[100]$. The resolved shear stresses on these two twin systems ($\tau_{(\bar{1}11)[11\bar{2}]}$ and $\tau_{(\overline{111})[2\bar{1}1]}$) are marked by the green and blue lines for each material model.

The maximum resolved shear stresses for each investigated crystal orientation and material model are presented in Figure 7. Here, HY stands for high yield stress, LY for low yield stress, HW for high work hardening behaviour, LW for low work hardening behaviour and noW for no work hardening behaviour. The influence of the material model taking into account plasticity can be clearly seen in Figure 7. A lower yield stress as well as less work hardening results in a lower resolved shear stress. The decrease of the resolved shear stress depends not only on the material model but also on the crystal orientation. One reason for this behaviour is the position of the resolved shear stress maximum. As of today, none of the models can perfectly describe the experimental results. This being said, some combinations come close, e.g., LYLW has the highest resolved shear stress on the experimentally identified twin systems within the two crystal orientations exhibiting twins, but the maximum resolved shear stress of other crystal orientations is higher. In contrast, LYnoW have the highest resolved shear stresses within the right crystal orientation, but within ND $[0\bar{1}1]$ SD $[100]$ not the experimentally identi-

fied twin system has the highest resolved shear stress. Here, it can be thought of an optimization of the work hardening behaviour. LYnoW and LYLW results into resolved shear stresses smaller than the critical resolved shear stress for twin formation. All the so far discussed material models are isotropic. Crystal plasticity FEM was used to consider elastic and plastic anisotropy and a material model without work hardening was chosen to be able compare to LYnoW. This comparison demonstrates that the resolved shear stresses of three out of the four investigated crystal orientations increase. The resolved shear stress values of the crystal orientations with twins are therefore closer to the critical resolved shear stress for twins in the literature. At the same time, the experimentally identified twin system of the crystal orientation ND $[0\bar{1}1]$ SD $[100]$ is not the twin system with the highest resolved shear stress and the resolved shear stress on the crystal orientation ND $[0\bar{1}1]$ SD $[0\bar{1}1]$ is too high compared to literature values. This demonstrates the sensitivity of the resolved shear stress on the material model in CP-FEM. Encouraged by these promising first results, we are considering writing a joint DFG proposal with Prof. Böhlke, to follow up much more systematically.

Tribological loading of poly and single crystalline CoCrFeMnNi typically led to the activation of twins on a maximum of one twin system. As we asking the question whether we might be able to activate more twin systems, we additionally investigated a material with a lower stacking fault energy. This material was found in CoCrNi in order to stay within the material family derived from the Cantor alloy CoCrFeMnNi. These experiments were conducted on coarse-grained samples with a 10 mm SiC sphere as the counter body. As can be seen in Figure 8, three twin systems were activated within the deformation layer. At the intersection of two twin systems, a displacement of one of the twins was observed. This allowed us to resolve the temporal sequence of the activated twin systems. Until we had published our results, there had been no consensus in literature on the temporal sequence of twinning on different systems. For us, it was clear that the barrier twin has to be displaced after being crossed by an incident twin as all twins exhibit a shear operation. Based on this line of thinking, the orange coloured twin system in Figure 8 was formed first, afterwards the red coloured one and at last the green coloured one were activated. With these model experiments, which can not be described here in all detail due to space restrictions, and together with the probing of the stress field with deformations twins itself, we are now at a point we– and the entire materials tribology community – are able to systematically test stress field models; an essential prerequisite to tailor materials and microstructure to tribological loading scenario in technical systems.

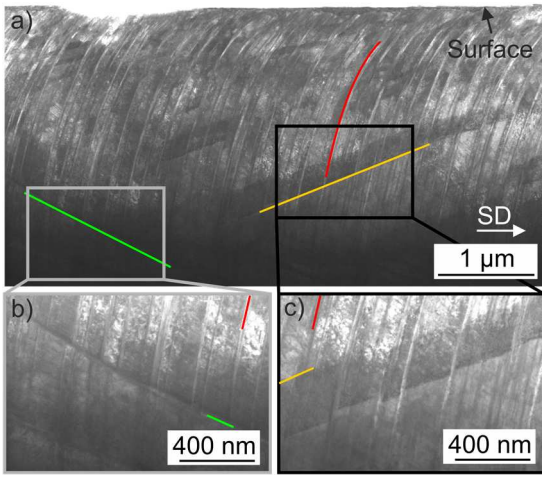

Figure 8. Deformation layer after a single trace experiment on CoCrNi.

Handling of research data

All data that was generated during this grant and that led to journal publications, was published on KIT's open data platform "KIT Open". There the data was associated with a doi, making it findable for other researchers; please see the list of publications below. All data generated within this grant is preserved long- term according to DFG regulations. We have also published an ontology for tribological experiments and are actively involved in the "Helmholtz Metadata Collaboration Platform (HMC)" through KIT's role within the Helmholtz Association.

[1] A. Dollmann, A. Kauffmann, M. Heilmaier, A. Srinivasan Tirunilai, L. S. Mantha, C. Kübel, S. J. Eder, J. Schneider und C. Greiner: *Dislocation-mediated and twinning-induced plasticity of CoCrFeMnNi in varying tribological loading scenarios*, Journal of Materials Science 57 (2022), S. 17448–17461.
[2] I. Karaman, H. Sehitoglu, K. Gall, Y. I. Chumlyakov und H. J. Maier: *Deformation of single crystal Hadfield steel by twinning and slip*, Acta Materialia 48 (2000), S. 1345–1359.
[3] H. Fujita und T. Mori: *A formation mechanism of mechanical twins in F.C.C. Metals*, Scripta Metallurgica 9 (1975), S. 631–636.
[4] T. Mori und H. Fujita: *Twinning Deformation in Single Crystals of Cu-8 at%Al Alloy*, Transactions of the Japan Institute of Metals 18 (1977), S. 17–24.
[5] S. Miura, J.-I. Takamura und N. Narita: Orientation dependence of flow stress for twinning in silver crystals, in Transactions of the Japan Institute of Metals, 1968, pp. 555.
[6] C. Greiner, Z. Liu, L. Strassberger und P. Gumbsch: *Sequence of stages in the microstructure evolution in copper under mild reciprocating tribological loading*, ACS applied materials & interfaces 8 (2016), S. 15809–15819.
[7] G. M. Hamilton: *Explicit Equations for the Stresses beneath a Sliding Spherical Contact*, Proceedings of the Institution of Mechanical Engineers, Part C: Journal of Mechanical Engineering Science 197 (1983), S. 53–59.
[8] A. Dollmann, C. Kübel, V. Tavakkoli, S. J. Eder, M. Feuerbacher, T. Liening, A. Kauffmann, J. Rau und C. Greiner: *Deformation twins as a probe for tribologically induced stress states*, Communications Materials 5 (2024), S. 1–10.
[9] G. Laplanche, A. Kostka, O. M. Horst, G. Eggeler und E. P. George: *Microstructure evolution and critical stress for twinning in the CrMnFeCoNi high-entropy alloy*, Acta Materialia 118 (2016), S. 152–163.
[10] C. Wagner und G. Laplanche: *Effect of grain size on critical twinning stress and work hardening behavior in the equiatomic CrMnFeCoNi high-entropy alloy*, International Journal of Plasticity 166 (2023), S. 103651.
[11] M. Kawamura, M. Asakura, N. L. Okamoto, K. Kishida, H. Inui und E. P. George: *Plastic deformation of single crystals of the equiatomic Cr−Mn−Fe−Co−Ni high-entropy alloy in tension and compression from 10 K to 1273 K*, Acta Materialia 203 (2021), S. 116454.
[12] I. V. Kireeva, Yu. I. Chumlyakov, Z. V. Pobedennaya, I. V. Kuksgausen und I. Karaman: *Orientation dependence of twinning in single crystalline CoCrFeMnNi high-entropy alloy*, Materials Science and Engineering: A 705 (2017), S. 176–181.
[13] A. Kareer, E. Tarleton, C. Hardie, S. V. Hainsworth und A. Wilkinson: *Scratching the surface: Elastic rotations beneath nanoscratch and nanoindentation tests*, Acta Materialia 200 (2020), S. 116–226.
[14] J. Zhu, Q. Zhou, Y. Huang, B. Zhou und J. Wang: *Surface deformation of single crystalline copper on different nano-scratching paths*, Journal of Materials Science 56 (2021), S. 10640–10652.
[15] Z. Wang, J. Zhang und J. Lu: *Effects of crystallographic orientations and grain boundaries on nanoscratching behaviour of unique bi-crystal Cu*, Wear 498–499 (2022), S. 204313.
[16] G. Laplanche, P. Gadaud, O. Horst, F. Otto, G. Eggeler und E. P. George: *Temperature dependencies of the elastic moduli and thermal expansion coefficient of an equiatomic, single-phase CoCrFeMnNi high-entropy alloy*, Journal of Alloys and Compounds 623 (2015), S. 348–353.
[17] K. Tanaka, T. Teramoto und R. Ito: *Monocrystalline elastic constants of fcc-CrMnFeCoNi high entropy alloy*, MRS Advances 2 (2017), S. 1429–1434.
[18] F. Otto, A. Dlouhỳ, C. Somsen, H. Bei, G. Eggeler und E. P. George: *The influences of temperature and microstructure on the tensile properties of a CoCrFeMnNi high-entropy alloy*, Acta Materialia 61 (2013), S. 5743–5755.

4 Published Project Results

4.1 Publications with scientific quality assurance

A. Dollmann, A. Kauffmann, M. Heilmaier, C. Haug and C. Greiner: Microstructural changes in CoCrFeMnNi under mild tribological load, Journal of Material Science 55 (2020) 12353. https://doi.org/10.1007/s10853-020-04806-0; raw data: https://doi.org/10.5445/IR/1000099798

A. Dollmann, A. Kauffmann, M. Heilmaier, A. Srinivasan Tirunilai, L.S. Mantha, C. Kübel, S.J. Eder, J. Schneider and C. Greiner: Dislocation-mediated and twinning-induced plasticity of CoCrFeMnNi in varying tribological loading scenarios, Journal of Material Science 57 (2022) 17448–17461. https://doi.org/10.1007/s10853-022-07661-3; raw data: https://doi.org/10.5445/IR/ 100039657

A. Dollmann, J.S. Rau, B. Bieber, L. Mantha, C. Kübel, A. Kauffmann, A.S. Tirunilai, M. Heilmaier and C. Greiner: Temporal sequence of deformation twinning in CoCrNi under tribological load, Scripta Materialia 229 (2023) 115378. https://doi.org/10.1016/j.scriptamat.2023.115378; raw data: DOI 10.5445/IR/1,000,152,864

A. Dollmann, C. Kübel, V. Tavakkoli, S. J. Eder, M. Feuerbacher, T. Liening, A. Kauffmann, J. Rau and C. Greiner: Deformation twins as a probe for tribologically induced stress states, Communications Materials 5 (2024), 1–10. https://doi.org/10.1038/s43246-023-00442-8; raw data: https://doi.org/10.35097/1732

H. J. Ehrich, A. Dollmann, P. G. Grützmacher, C. Gachot und S. J. Eder: Automated identification and tracking of deformation twin structures in molecular dynamics simulations, Computational Materials Science 236 (2024), 112878. https://doi.org/10.1016/j.commatsci.2024.112878

4.2 Other publications and published results

A. Dollmann, Dissertation, 2024, DOI: 10.5445/IR/1000170908

A. Dollmann, M. Feuerbacher, T. Liening, A. Kauffmann, J. S. Rau and C. Greiner: Unveiling the tribological stress field at high friction forces with deformation twins (in preparation)

A. Dollmann, A. Dyck, A. Kauffmann, T. Böhlke, C. Greiner: Tribological stress field model validation by using deformation twins as probes (in preparation)

4.3 Patents (applied for and granted)