Lamellar eutectic NiAl–Cr(Mo) alloys show an increased fracture toughness due to different toughening mechanisms. These mechanisms result from the fibrous or lamellar microstructure of the two constituting phases α-Cr(Mo) and β-NiAl. However, the fracture toughness of the individual phases and the evolution from early crack growth to the toughening mechanisms have not yet been systematically studied. Herein, bending tests on focused ion beam (FIB)-notched microcantilever beams are used to characterize the small-scale fracture properties. The micromechanical investigations reveal that the fracture toughness of the α-Cr(Mo) phase (7.5 – 9.1 MPa/m) is much higher than the fracture toughness of β-NiAl (2.2 – 2.9 MPa/m). Larger cantilevers in the crack arresting orientation show an enhanced fracture toughness with up to 14.4 MPa/m, which is still lower than the one of macroscopic experiments. This is attributed to the small interaction volume of the crack, which hinders the full exploitation of potential extrinsic toughening mechanisms.

1. Introduction

The intermetallic phase NiAl, which has a high melting point, good thermal conductivity, high oxidation resistance, and relatively low density,\textsuperscript{[1]} is an interesting material for high-temperature applications. However, its low fracture toughness and poor creep strength at high temperatures so far restrict its industrial applications as bulk material. Early work by Walter and Cline\textsuperscript{[2–6]} showed that eutectic systems of β-NiAl, where a solid-solution body-centered cubic (bcc) phase is embedded in the B2-ordered NiAl phase, exhibit improved fracture properties. Especially the addition of Cr and Mo increases the fracture toughness to values of 14.5 – 28.6 MPa/\(\sqrt{m}\)\textsuperscript{[7–10]} compared with 1.8 MPa/\(\sqrt{m}\) of pure NiAl when tested in the soft orientation and 6.9 MPa/\(\sqrt{m}\) in the hard orientation.\textsuperscript{[10]}

It is therefore assumed that interactions between propagating cracks and the ductile bcc phase improve the fracture resistance and ductility. This is enabled by directionally growing the eutectic alloys, which results in a highly aligned microstructure along the growth direction. For example, the morphology of stoichiometric NiAl alloys with 34 at.\% Cr consists of a NiAl matrix with Cr fibers, with a <100> orientation along the growth direction. If Cr is replaced with Mo, the growth direction shifts to <111> and a lamellar microstructure forms.\textsuperscript{[2,11]} The rather small dimensions of the order of \(\mu m\) of these refractory metal phases and the fact that they are nearly free of dislocations lead to their high strength, as demonstrated by micropillar compression tests.\textsuperscript{[2,13]}

The mechanisms which increase the fracture toughness were shown to be related to extrinsic toughening mechanisms such as crack trapping, crack deflection, interface debonding, and crack bridging.\textsuperscript{[12–14]} Those effects are dominant in the direction normal to the interphase boundaries, which is called crack arrest orientation. The fracture toughness has to be differentiated between the crack initiation toughness, which describes the beginning of crack growth in one phase of the composite and the increasing crack growth resistance or crack propagation toughness. This resistance increases, until it converges into the crack arrest toughness.\textsuperscript{[15,16]} As most studies so far have focused on macroscopic fracture properties, the microscopical influence of the individual phases and the change of the crack propagation toughness are not yet fully understood.

In this work, two directionally solidified NiAl–Cr(Mo) alloys with lamellar microstructures and different Mo contents were studied. The experiments were separated in two parts: 1) in situ microcantilever fracture tests were conducted to investigate the fracture toughness of the individual phases and 2) ex situ microcantilever tests were conducted to evaluate the behavior of the composites in the crack arresting direction as well as for comparison with macroscopic data.
2. Experimental Section

2.1. Materials

The NiAl–31Cr–3Mo (at.%) and NiAl–28Cr–6Mo (at.%) single-crystalline samples were fabricated via directional solidification in vacuum by the Bridgman method at Karlsruher Institut für Technologie, KIT; see the study by Kellner[17] for experimental details. The growth direction was <111> crystal orientation. The thickness of the lamellae varies with the Mo content and is 0.5 ± 0.3 μm for the α-Cr phase and 0.9 ± 0.2 μm for the β-NiAl phase in the 3 at.% Mo-containing alloy and 0.3 ± 0.1 μm and 0.6 ± 0.3 μm for the 6 at.% Mo-containing alloy, as shown in Figure 1. The used deviation for the results was standard deviation. Subsequently, the rod-shaped specimens were cut in small cubes with a diameter of 5 mm and ground with SiC paper. To obtain a deformation-free surface, they were additionally chemomechanically polished with OP-S suspension (Struers GmbH).

2.2. Microcantilever Fabrication

A focused ion-beam (FIB) milling system combined with a scanning electron microscope (SEM) (Helios NanoLab 600i, Thermo Fisher Scientific, MA, USA) was used to shape the notched cantilevers. The acceleration voltage of the gallium ions was kept constant at 30 kV. In a first step, the material around the beam was removed with a high current of 2500 pA, which was progressively reduced to 780 pA. The initial notch was milled with a very low current of 7 pA to minimize the beam radiation. This led to a notch radius of 10 nm, according to previous studies.[18,19] In a final step the surfaces were polished using 80 pA, which maintained a highly defined shape. The final cantilevers had an aspect ratio of 9:2:2 μm (L:length):W(width):T(thickness)) for the small beams, to test the properties of the individual phases, and 30:8:8 μm for the larger beams to study the properties of the composite. The notch length lₐ₀ was ≈0.4 μm. A cantilever with a notch positioned inside the NiAl phase is shown in Figure 1. For the determination of the single-phase fracture toughness, 4–10 microcantilevers were tested per phase and alloy. The fracture properties in the crack arresting orientation of the composites were tested by four microcantilevers per alloy. The lamellae thicknesses of the phases in the microcantilevers were within the standard deviation of the determined thicknesses, as mentioned in Section 2.1.

2.3. Testing Setups

Two different setups were used to determine the fracture properties of the samples. The first setup consisted of an instrumented micromanipulator (Kleindiek FMS, Kleindiek Nanotechnik, Germany) inside an SEM (1540EsB, Carl Zeiss, Germany), which was used to investigate the deformation behavior of the small microcantilevers in situ, as shown in Figure 1. The experiments had a displacement rate of ≈20 nm s⁻¹. For each phase in both alloys between five and nine cantilevers were tested. The notch was always placed in the middle of the phase with a minimum distance of 40% of the lamella thickness to the next interface. Individual fracture stages, like crack initiation and propagation, were directly observable. However, as the loading setup was unable to determine the displacement of the tip on the cantilever, a video had to be recorded to evaluate the displacement data by digital image correlation (DIC) (Software: VEDDAC 6.0, Chemnitzer Werkstoffmechanik, Germany). The micromanipulator was limited to a maximum force of 450 μN, which prevented its usage for larger beams. The second setup consisted of a nanoindenter G200 (KLA, CA, USA), which was used for ex situ testing of larger beams with a force of up to 500 mN. It also recorded more accurately force–displacement curves than the first setup. Furthermore, a better-defined contact with the beam

Figure 1. Secondary electron (SE) microstructure of the a) NiAl–31Cr–3Mo (at.%) and b) NiAl–28Cr–6Mo (at.%) alloy. The dark phase represents α-Cr, whereas the brighter one is β-NiAl. c) SE micrograph of an in situ microcantilever experiment. The initial notch is located in the α-Cr phase.
was established using a wedge-shaped diamond indenter tip. The wedge was previously aligned with the sample surface using a custom-made goniometer, which prevented undesired torsional torque and limited plastic indentation into the beam. A displacement rate of 20 nm s\(^{-1}\) was used, and a small oscillation (2 nm) was superimposed to the main loading. This enabled the continuous stiffness measurement (CSM) during the experiment,\(^\text{[20]}\) which made it possible to measure crack propagation, as previously demonstrated by Ast et al.\(^\text{[21]}\)

### 2.4. Fracture Toughness Evaluation

For brittle materials, linear-elastic fracture mechanics (LEFM) was applied and the following equation is used to calculate the fracture toughness \(K_{IQ,}\text{[22]}\)

\[
K_{IQ} = \frac{F_{\text{max}} L}{W^2 f(\frac{a}{W})}
\]

where \(F_{\text{max}}\) is the force at fracture. If the material response is elastic–plastic, the elastic fraction is calculated using the intersection of the force signal with 95% slope of the initial loading, which is then denoted as \(F_{0.95}\). \(L\) is the span length between the loading point and the notch, \(T\) is the beam thickness, \(W\) is the beam width, and \(a\) the crack length. \(f(a/W)\) is a dimensionless geometry function of the tested specimens calculated by Iqbal et al.\(^\text{[23]}\) with finite element (FE) simulations. However, the testing standard requirements\(^\text{[24,25]}\) for determining the plane strain fracture toughness \(K_{IC}\) cannot be fulfilled completely at this length scale (e.g., a fatigue precracking), we used the subscript “\(Q\)”, as suggested by Ast et al.,\(^\text{[26]}\) to describe the fracture toughness as “conditional” \(K_{IQ}\).

For the elastic–plastic fracture mechanics (EPFM), the fracture toughness was calculated via the \(J\)-integral, following the iterative method of the ASTM 1820.\(^\text{[25]}\)

\[
J_{(i)} = J_{(i)}^E + J_{(i)}^p
\]

\[
J_{(i)} = \frac{(K_{IQ,i})^2}{E} \left(1 - \nu^2\right)
+ \frac{f_{(i-1)}^p}{E} + \frac{\eta (A_{(i)} - A_{(i-1)})}{B(W - a_{(i-1)})} \left[1 - \frac{a_{(i)} - a_{(i-1)}}{W - a_{(i-1)}}\right]
\]

Here \(J^E\) and \(J^p\) are the elastic and plastic parts of the \(J\)-integral for iterative step \(i\). The elastic part is determined by Young’s modulus \(E\) and the Poisson’s ratio \(\nu\) with respect to the crystal orientation. The dimensionless number \(\eta\) is 2 for plane strain conditions\(^\text{[25]}\) and the plastic work \(A^p\) was computed numerically from the load–displacement curves for each point, subtracting the elastic work either by the initial loading slope \(k\) or by calculating it from the cantilever stiffness \(c = k^{-1}\).

As the orientation of the cantilevers was not determined prior to the experiments, the isotropic Young’s moduli and Poisson’s ratios, determined by Frommeyer et al.\(^\text{[27]}\) were used for the calculations. The Young’s modulus of \(\alpha\)-Cr was 286 GPa with a Poisson’s ratio of 0.22. The properties of \(\beta\)-NiAl were 171 GPa and 0.315, respectively. The influence of the Mo addition on the elastic properties was not considered and the elastic parameters were calculated with the rule of mixture for the large cantilevers. The area fraction in SEM images was used to determine a volume fraction of 46 ± 3% for \(\alpha\)-Cr in both alloys. Young’s modulus and the Poisson’s ratio of the composites were calculated as 224 GPa and 0.271. Dynamic measurements with CSM additionally allow for calculating the crack growth \(\Delta a\) from the decrease in contact stiffness, using a previous calibration via FE modeling.\(^\text{[26]}\) A continuous crack resistance curve can be generated by continuously recording the \(J\)-integral and the change in crack length. In addition to the determination of the critical \(J\)-integral \(J_{IQ}\), the onset of unstable crack growth can be determined. The fracture toughness was then calculated as follows.

\[
K_{IQ,J} = \sqrt{\frac{J_{IQ}E}{(1 - \nu^2)}}
\]

With the in situ setup, the lack of dynamic capabilities (i.e., CSM) did not allow to track crack growth. Instead the basic method was used to calculate the fracture toughness of the elastic–plastic cantilevers. This approach uses the plastic work up to the maximal applied force for the calculation of the \(J\)-integral. As previously shown by Ast et al.,\(^\text{[26]}\) its results correlate well with the ones determined by the crack resistance behavior for cantilevers with small plastic deformation.

### 3. Results

#### 3.1. Small Beams for Testing Individual Lamellae on the Composite

Four load–displacement curves for the \(\alpha\)-Cr(Mo) solid solutions and the \(\beta\)-NiAl phases of the 3 and 6 at.% Mo-containing alloys are shown in Figure 2a. A figure with all experiments is given in Figure S1, Supporting Information. The apparent differences between the curves of the same material originate in great part from small variations of the cantilever dimensions, which strongly impact their stiffness.

For better comparison, the stress intensity factor is shown in Figure 2b. As shown in Figure 2b, only the \(\beta\)-phases show brittle behavior. This justifies using the LEFM approach for evaluating the fracture toughness \(K_{IQ}\) from \(F_{\text{max}}\). In contrast, the \(\alpha\)-phase shows substantial strain hardening before fracture. After reaching a maximum, \(K_t\) slightly decreases due to stable crack propagation. As the curves do not correspond to a linear-elastic response, the fracture toughness has to be calculated with EPFM. The mean fracture initiation toughnesses of \(\beta\)-NiAl and \(\alpha\)-Cr of the 3 and 6 at.% Mo-containing alloys are shown in Table 1.

Fracture surfaces of \(\alpha\)-Cr and \(\beta\)-NiAl from the 6 at.% Mo-containing alloy are shown in Figure 3. The \(\beta\)-NiAl phase has a smooth fracture surface consisting of \{110\} cleavage planes, which are typical of fracture in the soft orientation.\(^\text{[28]}\) The in situ observations in the SEM show that stable crack growth occurred only for \(\approx 100\) nm in the \(\beta\)-NiAl phase before catastrophic failure. The stable crack growth region appears as the bright and rough area under the initial notch. Some fractured
cantilevers exhibit a rougher surface, indicating that the sample was oriented close to the hard orientation (Figure 3b), associated with significant plastic deformation. The corresponding fracture toughness is also higher, due to the longer stable crack growth. However, most of the samples appear to belong to soft orientation. In comparison, the α-Cr phase shows a more ductile fracture surface, as shown in Figure 3c). In situ SEM videos show that the experiments start with extensive crack blunting. Afterward, the crack starts to grow in a cleavage-like mode, yielding similar smooth surfaces as the soft orientation of β-NiAl. Regardless of this change in the fracture behavior, the crack growth remained stable and the cantilever did not break. Another difference is the high amount of small NiAl precipitates in the Cr phase, which were circumvented by the crack (see small dots in Figure 3c). Those precipitates can also be seen in Figure S3, Supporting Information.

3.2. Composite Fracture of Large Cantilevers

The fracture behavior of the multiphase cantilevers is shown in Figure 4a,b. Both alloys showed a similar load-displacement response with small amounts of plasticity. However, the 3 at.% alloy shows more plasticity, by a flattened slope, and longer stable crack growth. Both alloys exhibit similar maximum stress intensity factors of $4.6 \pm 0.5 \text{ MPa}\sqrt{m}$ for the 3 at.% Mo-containing alloy and $4.9 \pm 0.4 \text{ MPa}\sqrt{m}$ for the 6 at.% Mo-containing alloy. The scatter between the same alloys is quite low. It might be assumed that the notch intersected both phases, as the cellular growth caused the phases to not be perfectly parallel.

Figure 4c,d shows the crack resistance curves for both alloys. The crack propagation behavior of both alloys is different. The 3 at.% Mo-containing alloy shows a steeper slope in crack resistance, as well as higher-maximal $J$-integral values. This is supported by the higher plasticity shown in Figure 4. The crack initiation toughness is not practical, as it depends on the percentage of α-Cr and β-NiAl at the tip of the initial notch. Due to the nonlinear interface, this value cannot be extracted from the side view. Therefore, the crack propagation toughness at the maximal stable crack propagation was considered as the fracture toughness. The fracture toughness $K_{IQ,J}$ at critical failure ranges between $8.7 and 14.4 \text{ MPa}\sqrt{m}$ for the 3 at.% Mo-containing alloy and $4.9 \sim 12.4 \text{ MPa}\sqrt{m}$ for the 6 at.% Mo-containing alloy. In addition, the maximal stable crack growth of most cantilevers is higher in the 3 at.% Mo-containing alloy with $\Delta a < 0.4 \mu m$. Nevertheless, one of the 6 at.% Mo-containing alloy cantilevers shows a long stable crack growth for more than $1.6 \mu m$. This stabilization is attributed to the microstructure, which was analyzed postmortem.

The different fracture modes of the multiphase cantilevers are shown exemplarily for the 6 at.% Mo-containing alloy in Figure 5.

Table 1. Critical fracture toughness $K_{IQ,J}$ for the phases in the 3 and 6 at.% Mo-containing alloys.

|       | α-Cr     | β-NiAl   |
|-------|----------|----------|
| NiAl–31Cr–3Mo (at.%) | 9.1 ± 1.2 MPa $\sqrt{m}$ | 2.2 ± 0.7 MPa $\sqrt{m}$ |
| NiAl–28Cr–6Mo (at.%) | 7.5 ± 0.8 MPa $\sqrt{m}$ | 2.9 ± 0.8 MPa $\sqrt{m}$ |

Figure 3. SE micrographs of the fracture surfaces of the single-phase microcantilever experiments in the 6 at.% Mo-containing alloy. a) Soft orientation fracture surface of β-NiAl with cleavage planes. b) A hard orientation-like fracture surface of β-NiAl phase with a rough surface. c) Fracture surface of the α-Cr(Mo) phase with plastic deformation. Images of the tilted fracture surfaces are shown in Figure S2, Supporting Information.
One of the samples with lower fracture toughness and shorter stable crack growth, as shown in Figure 4b, reveals direct crack propagation in the plane of the notch (Figure 5a). The $\beta$-NiAl lamellae have a facet-rich surface with a low amount of plastically deformed areas, as already shown in Figure 3. Instead, the $\alpha$-Cr(Mo) phase deformed slightly plastically with a smooth fracture surface similar to the in situ experiments on the individual phase. As stable crack growth was only recorded for the first 0.4 $\mu$m, the influence of the later stages is not recorded during the measurement. This region is dominated by the passing of $\alpha$-Cr(Mo) and $\beta$-NiAl lamellae, as well as the crack deflection at the interface from the $\beta$-NiAl to the $\alpha$-Cr phase, where the crack finally stops.

The fracture surface of the tougher sample (Figure 5b) with more than 1.6 $\mu$m stable crack growth shows more lamellae crossings of the crack. The crack started growing in the initial direction but stopped at the first interface to the ductile $\alpha$-Cr(Mo) phase. As the crack cannot pass, it is deflected in another direction and renucleates beyond that phase, as shown in Figure 5c). After this, the crack growth becomes unstable and is not recorded by the stiffness signal. The further crack growth led to the formation of ligaments and bridging zones. Also, interface cracking took place, which reduces the stress intensity at the crack tip and stops the crack. The fracture behavior in both kinds of cantilevers is determined by crack arresting mechanisms, but stabilization starts with smaller crack growth in the cantilever with higher fracture toughness. Additional images of the fracture surfaces of both cantilevers are found in Figure S4 and S5, Supporting Information.

4. Discussion

4.1. Properties of the Individual Phases in the Composite

Fracture experiments using small-scale samples have the advantage that individual phases with small diameters, like eutectic alloys, can be tested and the effect of alloying elements on the individual phases can be studied. The obtained results on $\beta$-NiAl and $\alpha$-Cr(Mo) are compared with literature data and the multiphase cantilevers in Figure 6. The fracture toughness for NiAl reported in several studies depends strongly on the crystal orientation. In the soft orientation, it is between 1.8 and 3.5 MPa$\sqrt{\text{m}}$, whereas in the hard orientation, values between 5.1 and 6.9 MPa$\sqrt{\text{m}}$ have been reported.$^{[10,23,26]}$

The fracture toughnesses of $\beta$-NiAl in the 3 and the 6 at.% Mo-containing alloys are quite similar and fit well to other fracture toughness measurements of $\beta$-NiAl published for the soft orientation.$^{[26]}$ This is in good agreement with studies by Webler et al.,$^{[30]}$ which showed that the nonhard orientations have a nearly soft-oriented fracture toughness and that Cr addition reduces the fracture toughness. This results in the large scatter, which could be a result from a slightly different crystal orientation and an off-stoichiometric chemical composition. Recently, it has been shown that off-stoichiometric NiAl with an Al-rich...

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**Figure 4.** Calculated stress intensity factors versus the relative displacement until fracture of the multilayer cantilevers in the crack arrest orientation (inset) for a) the 3 at.% Mo-containing alloy and b) the 6 at.% Mo-containing alloy. The curves in (a) show additionally the unloading segments, which were needed to compensate the higher plasticity. The continuous crack resistance curves in c) the 3 at.% and d) the 6 at.% Mo-containing alloys.

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This could be a consequence of the simple replacement of Cr by Mo. Different studies have shown that this composition cannot be perfectly at the eutectic point, which would lead to changes of the stoichiometry.\textsuperscript{[32,33]} The change of the Mo addition from 3 to 6 at.% Mo does not seem to change the fracture toughness of the \(\alpha\)-\text{NiAl} phase significantly. Mo is known for increasing the ductility of \(\beta\)-\text{NiAl} at room temperature, but its solubility is low. Mo concentrations of more than 0.1 at.% do not have a further toughening effect.\textsuperscript{[34]} Therefore, \(\beta\)-\text{NiAl} is saturated with the maximum of 0.1% Mo in both alloys and should not be influenced by the higher Mo concentration.

In comparison with the \(\beta\)-\text{NiAl} phase, the \(\alpha\)-\text{Cr} phase has a four times higher fracture toughness and is the more ductile phase in both alloys.\textsuperscript{[29]} The determined fracture toughness of 7 – 10 MPa\(\sqrt{m}\) is in the range of the fracture toughness of pure (bulk) Cr.\textsuperscript{[35]} This shows that there is only small or no embrittlement of \(\alpha\)-\text{Cr} in NiAl–Cr(Mo) eutectic alloys. As both the solubilities of Ni and Al are quite low in \(\alpha\)-\text{Cr}, solid-solution hardening has only a minor effect. Due to processing by vacuum melting, no chromium nitrides formed, which normally decrease the fracture toughness of \(\alpha\)-\text{Cr} significantly.\textsuperscript{[36]} The orientation dependence on fracture toughness in chromium seems to be low. This is corroborated by the fracture surfaces, which do not indicate a preferred cleavage plane and reveal a homogeneously deformed structure.

The change from 3 to 6 at.% Mo lowers the fracture toughness of the \(\alpha\)-\text{Cr} phase from 9.1 ± 1.2 MPa\(\sqrt{m}\), to 7.5 ± 0.8 MPa\(\sqrt{m}\). As reported in the literature, Mo acts as a solid-solution hardener in \(\alpha\)-\text{Cr} but does not lead to embrittlement.\textsuperscript{[37]} Accordingly, the different fracture toughnesses can be additionally an effect of microstructural changes. The lamellae are thinner in the alloy with the higher Mo content. Hence, the thinner lamellae in the 6-Mo alloy limit the extension of the plastic zone around the crack tip. This could reduce the ductility of the material. Assuming that they are nearly defect free and have a strength of 1500 MPa as the fibrous eutectic \(\beta\)-\text{NiAl}–\(\alpha\)-\text{Cr(Mo)} alloys with lower amounts of Mo,\textsuperscript{[13]} the plastic zone can be calculated to have a size of about 3 \(\mu\)m. This size is bigger than the layer thickness in both alloys. Hence, the measured fracture toughness of \(\alpha\)-\text{Cr} must be seen as an intrinsic value of the lamella size but not as a bulk property.

Another effect of Mo addition is the promotion of precipitates in both phases, as shown by Johnson et al.\textsuperscript{[37]} The alloy with the higher Mo content had a much larger and a higher volume fraction of \(\beta\)-\text{NiAl} in the \(\alpha\)-\text{Cr} lamellae than the 3 at.% Mo-containing system. The amount of precipitates in the \(\beta\)-\text{NiAl lamellae was constantly lower than in the \(\alpha\)-\text{Cr} lamellae, as shown in Figure S3, Supporting Information. This is an effect of the higher lattice mismatch in the 6 at.% Mo-containing eutectic alloy, where the size of precipitates has to be larger to compensate for the internal stresses. The higher amount can be a result of a higher solubility of \(\beta\)-\text{NiAl} in the \(\alpha\)-\text{Cr} phase by the Mo addition. As shown in Figure 3a,b), the precipitates cannot be seen in the fracture surface images of \(\beta\)-\text{NiAl}. Hence, they seem to have no influence on the fracture toughness of the brittle phase. In \(\alpha\)-\text{Cr} (Figure 3c), the crack surrounded the precipitates, which are left at the crack surface. This suggests that the crack deflected at their surfaces. As \(\beta\)-\text{NiAl} and the interface are more brittle than \(\alpha\)-\text{Cr}, the fracture toughness presumably reduced. In addition, the larger obstacles reduce the mobility of dislocations and thereby decrease the fracture toughness.

Figure 5. SE micrographs of two 6 at.% Mo-containing microcantilevers in the crack arresting orientation after testing. a) Cantilever with the lowest fracture resistance. b) Cantilever with long stable crack growth and the highest fracture resistance. c) A higher-magnification image of the cantilever with the highest fracture resistance. d) A sketch of the toughening mechanisms, which were present in the cantilevers.

Figure 6. Fracture toughness of the individual phases in the 3 and 6 at.% Mo-containing alloys as well as the multiphase cantilevers. These are compared with the literature data of stoichiometric NiAl, tested by microcantilevers\textsuperscript{[21,26]} and macroscopic tests for pure Cr.\textsuperscript{[29]}

![Figure 5](https://www.aem-journal.com/2001464/6.png)

![Figure 6](https://www.aem-journal.com/2001464/7.png)
Anyhow, the fracture toughness of α-Cr is only mildly decreased in comparison with bulk Cr. In addition, the large error bar in the experiments only indicates that the mentioned embrittlement effects have an influence on the fracture toughness. Nevertheless, it is still rather high in comparison with the β-NiAl phases. As both phases have lower fracture toughnesses than the bulk NiAl-Cr composites, their combination and interaction with the crack are essential to the overall performance.

4.2. Properties of the Composites

To explain the fracture behavior and crack propagation in the composites, larger cantilevers, containing more than one phase in the crack arresting direction, were investigated. There is a large scatter in the crack propagation toughness at the end of the stable crack growth of the large cantilevers ranging from 4.9 MPa√m to 14.4 MPa√m.

The similarity in these experiments is that the multiphase cantilevers break after reaching the force maximum/Kmax, maximum, as shown in Figure 4. This is different to the failure of the ductile α-Cr cantilevers of Figure 2, which show stable crack propagation even after the maximum. This is a result of the different crack growth stages, which are visible in the fracture micrographs (Figure 5) of the multiphase cantilevers. During the start of the experiment, the notch is located either in the ductile α-phase, the brittle β-phase, or it intersects both phases, due to the curvy interface. If local stress exceeds the threshold for the propagation of the crack, the phase, in which the notch is located, starts to fail. This is influenced by the distance to the α/β interface, as the interface can affect the dislocation nucleation and motion. If the notch is in the brittle β-NiAl phase, it cleaves until it reaches the next brittle-to-ductile lamella transition. If the notch is located in the α-Cr lamella or if it intersects both phases, the crack grows stable, until it reaches the interface to the brittle β-NiAl. With the higher crack resistance of the ductile phase, the stress intensity is too high for the brittle β-NiAl, which cleaves until the crack tip also reaches the next ductile phase. Therefore, the first stage of the stable crack growth regime is limited by the size of the ductile phase. Hence the multiphase cantilevers with the lower fracture toughness in Figure 4 show a maximum stable crack propagation of 0.2–0.4 μm, which is within the range of the lamella size of both alloys. If the crack is not arrested at the interface to the ductile phase and passes through that phase, the stress intensity exceeds the fracture toughness of the following lamellae in the weaker cantilevers, which results in unstable failure. If, in contrast, the ductile phase behind the brittle β-NiAl lamella withstands the stresses, the crack is arrested and the cantilever has a longer stable crack growth with a higher crack growth resistance. Such a crack arrest is shown in Figure 5b) along the initial crack plane.

The further crack growth after crack arrest can be described by different mechanisms, as shown in Figure 5d). If the ductile lamella is able to deform, the crack will grow through it. Consequently, the crack will again meet the brittle β-NiAl, which will then also break in a brittle manner. This leads to cycles of discrete crack advances, whereby the ability of the ductile phase to arrest the crack is crucial for either stable crack growth or unstable failure. Nevertheless, this cyclic behavior is not the only possible mechanism for further stable crack growth. Figure 5 also shows that the crack can be deflected along the interface between α-Cr and β-NiAl. If the interface orientation leads to meandering of the crack, as shown in the crack arresting orientation of lamellar samples, the crack dissipates plastic energy for the longer crack path. As the crack deflection along the interface only occurs at the crack transition from the brittle to the ductile phase (Figure 5b), the interface strength seems to be higher than the stress necessary for the fracture of β-NiAl. Therefore, a weaker interface can increase the energy dissipation by enabling the deflection at both lamella crossings of the crack.

A third observed mechanism is the crack reorientation behind the ductile α-Cr phase, as shown in Figure 5c). If the crack cannot grow through the ductile phase or if it is deflected along the interface, stress can be transferred through the ductile phase to form a new crack in the next brittle β-NiAl lamella by crack reorientation. Hence, the crack passes around the remaining intact α-Cr lamella. If the new crack tries to further open, the remaining phase has to be deformed plastically; this leads to a crack closing force. This force reduces the stress at the crack tip and increases the fracture resistance. Consequently, all three mechanisms are important for stabilization of the crack in the more ductile multiphase cantilevers. Nevertheless, those mechanisms were mostly seen during unstable crack growth, as the compliance method for the calculation of the crack propagation measured only a stable crack growth of less than 1.7 μm. As shown in Figure 5, this only covers less than 50% of the final crack length. This restrained the quantification of those effects on crack growth resistance.

The crack resistance behavior shown in Figure 4 of both alloy systems reveals that the weakest cantilevers of the 3 at.% Mo-containing alloy have a higher J-integral at the maximal stable crack growth and exhibit prolonged crack length in comparison with the 6 at.% Mo-containing alloy. This is a consequence of the thicker lamella size in the 3 at.% Mo alloy. The crack can grow longer in a stable manner until it leaves the ductile phase. Therefore, the minimal crack growth for the 3 at.% Mo-containing alloy is longer before critical failure can occur. This only explains the differences for the weakest cantilevers, whereas the tougher cantilevers of Figure 5 with a maximal crack growth longer than the lamella size depend on the toughening mechanisms of crack deflection and reorientation. Nevertheless, the fracture toughness of the multiphase cantilevers is even for the toughest ones lower than 14.5 – 28.6 MPa√m of macroscopic experiments.

On the one hand, the different lamella thicknesses have a significant influence on the fracture toughness of microcantilevers. With a smaller thickness, the crack can only grow through the ductile phase until it reaches a brittle phase, where most of the measured samples fail by unstable crack growth. This is shown in Figure 5, as the 6 at.% Mo-containing alloy has a finer microstructure and lower fracture toughness at the failure of the cantilevers. As the lamella size only influences the early stages of crack growth, toughening mechanisms become more dominant with further crack growth. These are dominant at phase transitions and therefore a larger amount of interphase boundaries by smaller lamella sizes would increase the crack resistance. Therefore, the lower fracture toughness at crack initiation may be compensated. It can be concluded that a smaller lamella size would increase the fracture toughness. Electron beam melting might be an approach to control the lamella size, as done by
In addition, the fracture toughness of the β-NiAl phase seems to be lower than the interface strength. A ductilization of β-NiAl can increase the fracture toughness, as the toughening mechanisms can appear at both phase transitions of the crack. On the other hand, the dimensions of the multilayer cantilevers are too small to reach the full effect of the toughening mechanisms. As explained by Bloyer et al., there has to be a transition from crack initiation to an equilibrium state of failing and new reinforcing ductile lamellae, that bridge the crack. The multiphase cantilevers only contain a limited number of layers and, as shown in the fracture micrographs, only a few of them bridge the crack. Furthermore, the recorded stable crack growth is too small to contain a larger number of reinforcing phases. Thus, the fracture toughness in the microcantilever experiments is dominated by the crack initiation and only by a small extent through toughening mechanisms.

## 5. Conclusion

Microcantilever bending experiments show that the intrinsic fracture toughness of the individual phases of eutectic NiAl−Cr(Mo) alloys are similar to the fracture toughness of pure β-NiAl and α-Cr.

A higher Mo content (6 at.% Mo) results in the formation of smaller lamellae and promotes the presence of larger β-NiAl precipitates in the α-Cr phase. This results in a reduction of the fracture toughness of the ductile α-Cr (Mo) phase. The fracture toughness of the β-NiAl lamellae is not influenced by those changes.

For multiphase cantilevers with crack growth in the crack arresting orientation, the ductile phase determines the crack resistance at early stages. The overall fracture toughness of the composite cannot be determined at the macroscale, as the transition to a stable process zone at the crack front is not able to form within the dimensions of the cantilevers. Therefore, the large multiphase cantilevers had lower fracture toughness than macroscopic samples. However, toughening effects such as crack bridging, crack deflection, and renucleation are observable.

There is a correlation between the fracture toughness and the lamella size, presumably because smaller lamellae cause a larger fraction of toughening interfaces, which increases the fracture toughness on the macroscale. This effect is only observable in the crack arresting orientation.

### Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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### Conflict of Interest

The authors declare no conflict of interest.

### Data Availability Statement

Research data are not shared.

### Keywords

eutectic alloys, focused ion beam, fractures, intermetallic, micromechanics

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