



INHOMOGENEITY OF MISFIT STRESSES IN NICKEL-BASE SUPERALLOYS: EFFECT ON PROPAGATION OF MATRIX DISLOCATION LOOPS

Thomas Link, Alexander Epishin, Bernard Fedelich

► To cite this version:

Thomas Link, Alexander Epishin, Bernard Fedelich. INHOMOGENEITY OF MISFIT STRESSES IN NICKEL-BASE SUPERALLOYS: EFFECT ON PROPAGATION OF MATRIX DISLOCATION LOOPS. Philosophical Magazine, 2009, 89 (13), pp.1141-1159. 10.1080/14786430902877810 . hal-00514017

HAL Id: hal-00514017

<https://hal.science/hal-00514017>

Submitted on 1 Sep 2010

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



INHOMOGENEITY OF MISFIT STRESSES IN NICKEL-BASE SUPERALLOYS: EFFECT ON PROPAGATION OF MATRIX DISLOCATION LOOPS

Journal:	<i>Philosophical Magazine & Philosophical Magazine Letters</i>
Manuscript ID:	TPHM-09-Jan-0033.R1
Journal Selection:	Philosophical Magazine
Date Submitted by the Author:	05-Mar-2009
Complete List of Authors:	Link, Thomas; Technical University Berlin, Institute for Material Science and Engineering, BH 18 Epishin, Alexander; Technical University Berlin, Institute for Material Science and Engineering, BH 18 Fedelich, Bernard; Federal Institute for Materials Research and Testing, Department V.23
Keywords:	creep, dislocations, superalloys
Keywords (user supplied):	misfit stresses



Dr. Thomas Link (Corresponding Author)
1 Technical University Berlin, Institute for Material Science and Engineering, BH 18,
2 Ernst Reuter Platz 1, 10587 Berlin, Germany
3 Tel: +493031426175
4 Fax: +493031422996
5 E-mail: Thomas.Link@TU-Berlin.de

6
7 Dr. Alexander Epishin
8 Technical University Berlin, Institute for Material Science and Engineering, BH 18,
9 Ernst Reuter Platz 1, 10587 Berlin, Germany
10 Tel: +493078981
11 Fax: +493031422996
12 E-mail: alex.epishin@yahoo.de

13 Dr. Bernard Fedelich
14 Federal Institute for Materials Research and Testing, Division V.2
15 Unter den Eichen 87, 12205 Berlin, Germany
16 Tel: +493081043104
17 Fax: +493081041527
18 E-mail: bernard.fedelich@bam.de

19
20 Number of words in the manuscript is 5322
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

INHOMOGENEITY OF MISFIT STRESSES IN NICKEL-BASE SUPERALLOYS:
EFFECT ON PROPAGATION OF MATRIX DISLOCATION LOOPS

By T. Link^{1*}, A. Epishin¹ and B. Fedelich²

¹Technical University Berlin, Institute for Material Science and Engineering, BH 18,
Ernst Reuter Platz 1, 10587 Berlin, Germany

²Federal Institute for Materials Research and Testing,
Unter den Eichen 87, 12205 Berlin, Germany

Abstract

It is shown experimentally that during annealing and creep under low applied stresses the matrix dislocation loops frequently cross glide. The periodic length of the zigzag dislocations deposited in the interfaces is equal to that of the γ/γ' -microstructure. Initially the zigzag dislocations move in the (001) interface by a combination of glide and climb but then they stop near the γ' -edges and align along $\langle 100 \rangle$. Reactions of such dislocations lead to the formation of square interfacial networks consisting of $\langle 100 \rangle$ oriented edge dislocations. The complex dislocation movement is explained by the inhomogeneity of the misfit stresses between γ - and γ' -lattices. The tensile components of the stress tensor drive the dislocations through the channel, while the shear components near the γ' -edges cause the zigzag movement and the $\langle 100 \rangle$ alignment. The total effect is the most efficient relaxation of the misfit stresses. The results are relevant especially for single-crystal superalloys of the newest generations, which have an increased γ/γ' -misfit due to the high level of refractory elements.

Keywords: Superalloys, Creep, Dislocations, Misfit stresses

* Corresponding author.

1

2

3

4

5

6

7

8

9

10

11

12

13

14

15

16

17

18

19

20

21

22

23

24

25

26

27

28

29

30

31

32

33

34

35

36

37

38

39

40

41

42

43

44

45

46

47

48

49

50

51

52

53

54

55

56

57

58

59

60

1. Introduction

In nickel-base superalloys the interfacial area between γ -matrix and cuboidal γ' -precipitates is very high with an area density of about $10^7 \text{ m}^2/\text{m}^3$. Therefore the interface plays a crucial role in the structural and mechanical behaviour of superalloys. During the primary stage of high temperature creep the coherency stresses between γ - and γ' -phase are relaxed by interfacial dislocation networks, a process which is designated as loss of coherency of the γ/γ' -interfaces. The formation mechanism of these networks was extensively studied by [1-9], with the unanimous conclusion, that during primary creep $a/2\langle 110 \rangle$ dislocation loops are gliding on $\{111\}$ -planes through matrix channels. At the intersection between $\{100\}$ -interface and glide plane they leave behind $\langle 0\bar{1}1 \rangle$ oriented dislocation segments. Cross slip of the leading segment of a matrix dislocation loop generates zigzag-shaped interfacial dislocations.

Fig. 1 gives an impression of the resulting dislocation arrangement in the γ/γ' interfaces of CMSX-4 after creep-deformation at elevated temperature and high stress. Here dislocation loops can be recognized, having about the width of the matrix channel and a direction diagonal to the $\{100\}$ habit planes of the γ' -cubes, which is in full agreement with the described mechanism. However, when temperatures become high and stresses low, the appearance of the interfacial dislocations changes significantly (Fig. 2).

The dislocations now mostly lie parallel to the edges of the γ' -cubes and can hardly be recognised as trailing segments of dislocation loops. The aim of our investigations was to identify the mechanism responsible for the formation of such configurations.

E-mail address: Thomas.Link@TU-Berlin.de

2. Experimental

To obtain an overview of the process of loss of coherency during primary creep, four single crystal superalloys, SRR99, CMSX-4, CMSX-10 and TMS138 were investigated, representing generation 1 to 4 in superalloy development. Table 1 shows the increasing content of refractory elements and finally the use of ruthenium in the latest alloy. In the table the constrained misfit is given, as measured in the coherent γ/γ' compound in the Primary Dendrite Arm (PDA). The misfit of the isolated phases, the unconstrained misfit, is about two times smaller [13]. The misfit averaged over the whole dendritic structure is about 50-70% of the misfit in the PDA [10].

At high temperatures around 1100°C superalloys are usually tested for creep under applied stresses of about 100 MPa, which are comparable with misfit stresses. The applied stresses in the hottest section of a turbine blade are even lower. This means that both applied stress and misfit stresses become relevant for the dislocation movement in the matrix channels and γ/γ' -interfaces. To distinguish the effect of misfit and applied stress, dislocation structures obtained during creep tests were compared with those after annealing experiments. Table 2 gives an overview of the creep and annealing tests, presented in this paper. For all creep tests the load axis is [001].

The dislocation structures were investigated mainly by TEM (Philips, CM 30). Additionally a high resolution SEM was used (Leo Gemini 1530 VP), allowing to visualise interfacial dislocations via the grooves, generated by the dislocations on the γ/γ' -interface [14].

The spatial distribution of misfit stresses in the γ - and γ' -phases was computed by the Finite Element Method (FEM) using the software ABAQUS.

3. TEM results: Microstructural investigations

Fig. 3 shows a typical dislocation configuration in TMS-138 after creep at 1100°C, 137 MPa for 2h.

The micrograph illustrates the following principles:

• Interfacial dislocations are generated by dislocation loops, propagating in channels lying Perpendicular to the load axis ("P-channels").

• The average propagation direction of a loop is $\langle 100 \rangle$.

• The tip of the loop is narrow but with increasing distance from the tip the loop becomes wider.

• Two types of cross-slip can be distinguished: when the tip of the loop approaches a channel parallel to the average propagation direction ("R-channel") it is Repelled by cross slip, but when it impinges a channel perpendicular to the propagation direction ("C-channel") it Crosses the channel and cross-slips.

• The trailing zigzag line moves towards the γ' -edge, which is parallel to the average propagation direction. Along this edge it straightens in $\langle 100 \rangle$ direction.

Detailed inspection of the shape of the dislocations crossing the C-channels shows, that cross slip takes place directly at the γ' -edges (Fig. 4). Which side of the C-channel is selected, depends on the dislocation line direction (arrows in Fig.4).

Fig. 5a shows dislocation loops in SRR99 creep deformed at 980°C, 200 MPa, 45.4h, 0.3%. The beam direction is $\vec{k}_0 = [001]$. The loop under discussion is marked by arrows 1. It has the line vector

$\vec{\xi} = \frac{1}{\sqrt{2}}[\bar{1}10]$ and the Burgers vector $\vec{b} = \pm a/2[0\bar{1}1]$, so the glide plane is (111). The average

propagation direction is [100]. $\vec{b}$ has no component in the average propagation direction. This was found for all loops in all four superalloys, so it can be regarded as a general result.

Fig. 5b shows the foil tilted in $[\bar{1}01]$ orientation, so the (111) plane is now edge on. In deed the tip of the loop now appears as a line (arrows 1). Further on the left, where the loop has widened, the opposite interfacial segments are no longer lying in a common (111) plane (arrows 2). This means they have drifted by a nonconservative glide/climb movement on the lower and upper γ/γ' interfaces in opposite directions. The dislocation segments with [100] line vector give with $[100] \times [0\bar{1}1]$ pro forma (011) glide planes, but this is just a result of changing their original line directions from [110] and $[\bar{1}10]$ into [100] by interfacial glide/climb. So Fig. 5 confirms, that dislocation loops with $\vec{b} = a/2\langle 110 \rangle$ are gliding on

$\{1\bar{1}1\}$ planes through matrix channels. Their trailing components are lying in the lower and upper interfaces respectively. The Burgers vector has a component in stress direction and the resulting interfacial dislocations have 60° character. Dislocations with $\langle 100 \rangle$ line directions and edge character result from interfacial movement.

Especially when no stress is applied, the effect of alignment of the interfacial dislocations in $\langle 100 \rangle$ direction is very pronounced. This leads to dislocation networks, which are square rather than hexagonal. Inspection of literature shows, that interfacial dislocations with $\langle 100 \rangle$ line direction and square network meshes are found typically, when temperatures are very high and stresses low, or when specimen are simply annealed (Table 3).

Fig. 6 shows such networks in SRR99, annealed at 1100°C for 300 h. Fig. 6a is a TEM micrograph and Fig. 6b a SEM image of the same specimen. The TEM image visualises the network with a somewhat better resolution, whereas the SEM image supplies a better overview, but in any case the observed dislocation structures are practically the same.

The transition of $\langle 110 \rangle$ oriented interfacial dislocations into square networks can be recognized in Fig. 7, taken with a foil of TMS138, 1100°C , 137MPa, 2 h, 0.04%.

4. FEM results and discussion

4.1. FEM results for coherency misfit in CMSX-4 at 1100°C

During primary creep under uniaxial tensile load applied in $[001]$ direction the matrix dislocation loops cross glide in the P-channels (001). Driving forces for this movement are the applied stress σ_{33}^a and the misfit stresses $\hat{\sigma}^m$, given as 3D stress tensor. In this case the applied stress σ_{33}^a is quite uniformly distributed within the γ/γ' -microstructure because the difference of the elastic constants of γ - and γ' -phase

is not large [15, 16]. Therefore the stress σ_{33}^a causes just glide on one plane but not the cross glide on another.

The misfit stresses $\hat{\sigma}^m$ however differ strongly in the γ - and γ' -phases and follow the periodicity of the γ/γ' -microstructure. So the cross glide must be a consequence of local changes of the misfit stresses. To understand this effect the spatial distribution of $\hat{\sigma}^m(x, y, z)$ was calculated by FEM at 1100°C. The elastic constants of γ - and γ' -phases were taken to be the same and equal to those of CMSX-4 at 1100°C, i.e. $c_{11}=153$ GPa, $c_{12}=100$ GPa and $c_{44}=89$ GPa [17]. The unconstrained γ/γ' -misfit in CMSX-4, -0.25%, was obtained by extrapolation of neutron diffraction data from [13] up to 1100°C. The γ' -volume fraction 63% was measured on SEM images showing the γ/γ' -microstructure of CMSX-4 creep tested at 1100°C. It was assumed, that the corners of the γ' -cubes are slightly rounded with a radius r . The ratio between r and γ' -edge length l , $r/l \approx 20$, was measured by processing of dark field TEM images of undeformed CMSX-4. A 1/8th of the periodic cell of the γ/γ' -microstructure was represented by a mesh with 671151 elements: 388157 elements for the γ' -phase, the rest for the γ -phase. The structural model for the FEM calculations is shown in Fig. 8.

Fig. 9 represents components of the misfit stress tensor in two cross cuts along the P-channel (see Fig. 8). The normal stresses σ_{22}^m and σ_{33}^m are shown for a (001) plane lying in the middle of the channel (0.5 of the channel width w , Fig. 9a, b), the shear stresses σ_{12}^m and σ_{13}^m directly above the γ/γ' -interface (0.03 w). The area, where the γ' -cube is lying below the matrix is marked by a dashed line and designated as γ/γ' . It follows from the FEM calculations that the normal stress σ_{22}^m (Fig. 9a) is compressive in the γ/γ' area with a high absolute value of about 250 MPa. In the (010)-channel however it changes its sign. The normal component σ_{33}^m is tensile (Fig. 9b) but its value is much lower, namely below 50 MPa. The shear components σ_{13}^m and σ_{12}^m vanish in the middle of the channel but they can reach high values close to the edges of the cube. In Fig. 9 one can see, that σ_{13}^m has a maximum of about 250 MPa sharply confined to the (100) edge of the γ/γ' -area. Such an inhomogeneous distribution of shear stresses along interfaces is well-known from the mechanics of composite materials. It is called “end-effect” and occurs, e.g. in

composites reinforced by discontinuous fibres [e.g. 18]. The shear stress σ_{12}^m has also a local maximum at the γ' -corner, but it is not pronounced. It follows from the FEM results that the relevant components of the misfit tensor are σ_{22}^m and σ_{13}^m , while the components σ_{33}^m and σ_{12}^m are small and therefore less important for the dislocation movement. The distribution of the components σ_{11}^m and σ_{23}^m follow from those for σ_{22}^m and σ_{13}^m by permuting the directions 1 and 2. They are not shown here, because they don't play a role for the following considerations.

4.2. Forces acting on the dislocation loop

Fig. 10 shows a dislocation, which has a 90° kink close to the R-channel. The leading segment glides in the matrix, the trailing segments are lying in an upper and a lower γ/γ' -interface. The Burgers vector is

$$\vec{b} = \frac{a}{2} [0 \bar{1} 1].$$

The kink forms when the matrix segment cross glides from a $(\bar{1} 11)$ matrix plane to a (111)

plane. The leading dislocation is assumed to be the shortest segment connecting the lower and upper (001) interface along the $\{111\}$ glide plane. This gives the normalised line vectors $\vec{\xi} = \frac{1}{\sqrt{6}} [1 \bar{1} 2]$ on

$(\bar{1} 11)$ and $\vec{\xi} = \frac{1}{\sqrt{6}} [\bar{1} 1 2]$ on (111) . The average movement direction is $[100]$.

The gliding component of the elastic force acting on the dislocation is (see Appendix):

$$\vec{F}^{glide} = \tau b \vec{n} \times \vec{\xi} \quad (1)$$

where τ is the resolved shear stress (RSS), $b = |\vec{b}|$ and $\vec{n}$ the glide plane normal. The RSS is given by:

$$\tau = \left[\hat{\sigma} \left(\vec{b} / b \right) \right] \vec{n} \quad (2)$$

where $\hat{\sigma}$ is the stress tensor.

It should be mentioned that $\vec{n}$ appears in both equations (1) and (2), therefore the sign of $\vec{n}$ is irrelevant.

The signs of $\vec{b}$ and $\vec{\xi}$ however determine the sign of $\vec{F}^{glide}$. They have to be defined in such a way, that the edge component of a dislocation segment lying in the interfaces relaxes the misfit stresses of an alloy

having a negative misfit. According to the discussion in [19] for a negative misfit and a Burgers vector

$\vec{b} = \frac{a}{2}[0\bar{1}1]^1$, the dislocation segment in Fig. 10 lying in the lower interface must have the line vector $[110]$ on the $(\bar{1}11)$ glide plane or to $[1\bar{1}0]$ on (111) . In the upper interface the line vectors have opposite directions.

Applying (1) for the considered glide systems one gets on the $(\bar{1}11)$ and (111) planes respectively:

$$\vec{F}_{(\mp 111)}^{glide} = \tau_{(\mp 111)} \frac{a}{\sqrt{2}} \frac{1}{\sqrt{3}} [\mp 111] \times \frac{1}{\sqrt{6}} [\pm 1, -1, 2] = \tau_{(\mp 111)} \frac{a}{2} [1, \pm 1, 0] \quad (3)$$

The total RSS τ^{total} is the sum of the RSS τ^a , caused by the applied stress σ^a and the RSS τ^m caused by the misfit stress $\hat{\sigma}^m$.

$$\tau_{(\mp 111)}^{total} = \tau_{(\mp 111)}^a + \tau_{(\mp 111)}^m \quad (4)$$

When σ^a is applied uniaxially in $[001]$ direction, equation (2) gives for $\tau_{(\mp 111)}^a$:

$$\tau_{(\mp 111)}^a = \frac{1}{\sqrt{6}} \sigma_{33}^a \quad (5)$$

$\tau_{(\mp 111)}^a$ is homogeneously distributed and equal for both glide systems. For $\tau_{(\mp 111)}^m$ (2) gives:

$$\tau_{(\mp 111)}^m = \frac{1}{\sqrt{6}} (\pm \sigma_{12}^m \mp \sigma_{13}^m - \sigma_{22}^m + \sigma_{33}^m) \quad (6)$$

$\tau_{(\mp 111)}^m$ is inhomogeneously distributed. The difference between $\tau_{(111)}^m$ and $\tau_{(\bar{1}11)}^m$ is due to the opposite signs of the shear stresses σ_{13}^m and σ_{12}^m :

$$\tau_{(111)}^m - \tau_{(\bar{1}11)}^m = \frac{2}{\sqrt{6}} (-\sigma_{12}^m + \sigma_{13}^m) \approx \frac{2\sigma_{13}^m}{\sqrt{6}} \quad (7)$$

According to Fig. 9c, d the difference is caused nearly completely by σ_{13}^m , while the effect of σ_{12}^m is negligible.

Formula (6) can be used to attain a descriptive understanding how plastic deformation works on a microscopic level. The plastic strain tensor $\hat{\epsilon}$ produced by a dislocation gliding on a plane $\vec{n}$ over an area A is:

$$\hat{\varepsilon} = \frac{1}{2V}(\vec{b} \otimes \vec{A} + \vec{A} \otimes \vec{b}) \quad (8)$$

V is the crystal volume and $\vec{A} = A \cdot \vec{n}$. In our case $\vec{b}$ is $\frac{a}{2}[0\bar{1}1]$ and $\vec{A} = \frac{1}{\sqrt{3}}[\mp 111]$, which gives:

$$\hat{\varepsilon}(\mp 111) = \frac{a}{2\sqrt{3}V} \begin{pmatrix} 0 & \pm 1/2 & \mp 1/2 \\ \pm 1/2 & -1 & 0 \\ \mp 1/2 & 0 & 1 \end{pmatrix} \quad (9)$$

It follows, that glide on both octahedral planes causes a contraction ε_{22} perpendicular to the stress axis and an elongation ε_{33} along the axis. The shear strains ε_{12} and ε_{13} however have opposite signs for (111) and ($\bar{1}11$) glide, which means that they annihilate, when the loop cross glides in such a way, that a [100] average direction results. For this average movement the normal stresses σ_{22}^m and σ_{33}^m are important, whereas the shear stresses σ_{12} and σ_{13} determine locally, on which glide plane the dislocation propagates.

The RSS $\tau_{(111)}^m$ calculated according to (6) is shown in Fig. 11. In order to clarify how the C-channel is crossed not only the area of the C-channel but also of the neighbouring γ' -cubes is analysed. The RSS $\tau_{(111)}^m$ depends very sensitively on the distance between the (001) section and the (001) γ/γ' -interface. Therefore sections in 3 distances are shown: in the middle of the P-channel $0.5w$ (a), very close to the (001) interface $0.03w$ (c) and in-between $0.25w$ (b).

Fig. 11 shows immediately, that a dislocation arriving from the left side on a (111) plane, is rejected when approaching the R-channel ($\tau_{(111)}^m$ negative) but attracted, when it comes to the edge of the cube at the C-channel. ($\tau_{(111)}^m$ positive). Quantitative information about the distribution of $\tau_{(111)}^m$ in both channel types is given by Fig. 12: in Fig. 12a from the center of one γ' -cube, crossing the R-channel (010) and ending in the center of the next cube, in Fig. 12b the same across the C-channel. For Fig 12a RSS was averaged for the distances $0.5w$, $0.25w$ and $0.03w$ along the matrix dislocation segment. From the cubical symmetry follows that σ_{13}^m and σ_{12}^m vanish along the (100) plane through the cube center (see also Fig. 9c, d).

¹ The sign of $\vec{b}$ is defined according to the so-called FS/RH convention [20].

According to equation (7) the difference between $\tau_{(111)}^m$ and $\tau_{(\bar{1}\bar{1}\bar{1})}^m$ is caused by these components. The line however, along which RSS in Fig. 12a is calculated lies in this plane and therefore in this case $\tau_{(111)}^m = \tau_{(\bar{1}\bar{1}\bar{1})}^m$.

We now discuss the forces acting on the leading screw dislocation of the $\frac{a}{2}[0\bar{1}1](111)$ glide system. The distributions of $\tau_{(\bar{1}\bar{1}\bar{1})}^m$ can be obtained by mirroring Figs 11 and 12b on the (100) plane.

From Figs 9, 11 and 12 follows:

• Inside the γ/γ' area the RSS is positive. Fig. 11 and Figs. 12a, b show, that RSS is nearly constant across the P-channel in [001] direction and along this channel on the (001) plane. According to equation (6) and Fig. 9 RSS it is mostly caused by σ_{22}^m . The RSS increases when tensile load is applied (arrows in Fig. 12a). Applied and misfit stress σ_{33}^a and σ_{22}^m drive the dislocation loops through the P-channel, thus elongating the specimen by ε_{33} as well as relaxing the misfit stresses by ε_{22} (see equation (9)).

• Close to the R-channel (010) $\tau_{(\bar{1}\bar{1}\bar{1})}^m$ strongly decreases and becomes slightly negative in the center of the R-channel (Fig. 12a). The change of $\tau_{(\bar{1}\bar{1}\bar{1})}^m$ is mostly caused by σ_{22}^m (Fig. 9). In general the matrix is under compression to attain coherency with the γ' -phase. Locally however it is under tension, namely when the P-channel crosses the R-channel.

• Crossing the C-channel (100) $\tau_{(\bar{1}\bar{1}\bar{1})}^m$ shows a strong oscillation near the (100) interface (Fig. 12b). From Fig. 9 follows, that the main reason is σ_{13}^m . The shear stress piles up in the γ/γ' -area, when approaching the C-channel. It has opposite directions on the left and right side of the C-channel, which explains the oscillation of RSS in this region.

4.3. Cross glide at the R-channels (010)

From the zigzag shape of the deposited interfacial dislocations (see Fig. 3) follows, that the matrix segment always changes its glide plane when approaching the R-channel (010). This type of cross slip is

schematically shown in Fig. 10. From Fig. 12a follows, that the average RSS rapidly decreases from about 120 MPa down to about -10 MPa when the dislocation moves on the $(\bar{1}11)$ glide plane from the center of the γ/γ' -area to the middle of R-channel. It is important that in a good approximation $\tau_{(111)}^m$ is equal to $\tau_{(\bar{1}11)}^m$. This is exactly correct for $x=0$ but also well achieved nearly along the complete R-channel, because the difference $\tau_{(111)}^m - \tau_{(\bar{1}11)}^m$ is proportional to σ_{13}^m (equation (7)). In this area however σ_{13}^m is practically zero according to Fig. 9c. So, the shear stress on the dislocation continuously decreases, when it approaches the R-channel on $(\bar{1}11)$, but the force increases as soon as the dislocation cross slips into (111) and thus returns back from the edge to the centre of the γ/γ' -area. This misfit stress barrier at the R-channels can be overcome if the applied stress σ_{33}^a is strong enough as observed during creep under high applied stress at lower temperatures (Fig. 1).

4.4. Cross glide at the C-channels (100)

TEM analysis of propagating dislocation loops shows that a loop cross glides every time when crossing a C-channel (100). It is remarkable that this bend in the dislocation line takes places only on one of the two interfaces of the C-channel (100) as observed in Fig. 3 and schematically drawn in Fig. 13.

For a detailed discussion the distribution of $\tau_{(111)}^m$ in the (010) section across the C-channel (100) was plotted (Fig. 12b). The RSS $\tau_{(\pm 111)}^m$ consists of the components σ_{12}^m , σ_{13}^m , σ_{22}^m and σ_{33}^m (see formula (6)).

The normal stresses σ_{22}^m and σ_{33}^m do not depend on the sign of the x -component of $\vec{n}$ and σ_{12}^m is zero along $y=0$ (Fig. 9d). As mentioned σ_{13}^m has opposite signs on the left and right side of the C-channel.

This means $\tau_{(111)}^m$ and $\tau_{(\bar{1}11)}^m$ are mirror symmetric with the symmetry plane lying in the middle of the C-channel. For a better understanding of the effect of $\tau_{(111)}^m$ on the movement of the matrix dislocations, $\tau_{(111)}^m$ is given as a colour map on a longitudinal (010) cut through the middle of four neighbouring γ' -cubes (Fig. 14). The leading part of the dislocation loop, which is actually curved, is approximated by the

shortest connection between the trailing dislocation segments lying in the upper (U) and lower (L) (001)-interface. When the lower part of the loop reaches the channel crossing, it extends under the influence of the local maximum of $\tau_{(111)}^m$ towards the opposite channel side (dotted line in Fig. 14), where $\tau_{(111)}^m$ strongly decreases. Because of the mirror symmetry, here $\tau_{(\bar{1}11)}^m$ has its maximum. Therefore this leading dislocation tip turns into screw orientation and cross slips from (111) to $(\bar{1}11)$. The upper part of the dislocation loop follows the leading tip into the new $(\bar{1}11)$ glide plane.

4.5. Alignment of the interfacial dislocations parallel to the R-channels (010)

The experiment shows that the deposited zigzag interfacial dislocations first move in the (001) interface by glide/climb but then stop near the γ' -edge, where they align in $\langle 100 \rangle$ direction parallel to the R-channels (see Figs 2 and 3). To analyse the interaction of the kink, schematically shown in Fig.10, with the misfit stresses at the γ' -edge, the elastic force driving a straight dislocation segment with $\vec{b} = \frac{a}{2}[0\bar{1}1]$ was calculated:

$$\vec{F}^{el} = [(\hat{\sigma}^a + \hat{\sigma}^m)\vec{b}] \times \vec{\xi} \quad (10)$$

The line direction was approximated by $\vec{\xi} = [100]$, which considers the change of the shape of the kink: the sharp bend of the dislocation line becomes trapezoidal. Under uniaxial tension in [001] direction the component $F_{[010]}$ of the elastic force is:

$$F_{[010]} = \frac{a}{2}(\sigma_{33}^a - \sigma_{23}^m + \sigma_{33}^m) \quad (11)$$

One can see from (11) that on the (001) interface the dislocation is driven by the applied stress σ_{33}^a as well as by the misfit stresses σ_{23}^m and σ_{33}^m . The normal stress σ_{33}^a is positive and homogeneously distributed, so it moves the dislocation always forward. The normal stress σ_{33}^m supports the applied stress but its contribution is small. The shear stress σ_{23}^m shows a different behavior: it is generally very small but

has a high maximum at the transition to the R-channel (010). (The distribution of σ_{23}^m can be obtained from that for σ_{13}^m by permuting the directions 1 and 2). Fig. 14 shows the distribution of $F_{[010]}(\hat{\sigma}^m)$ for $\sigma_{33}^a = 0$. From Fig. 15 follows, that inside the γ/γ' -area $F_{[010]}(\hat{\sigma}^m)$ is positive and relatively small, about 50 MPa. But due to the sharp peak of σ_{23}^m just before the R-channel it becomes strongly negative (below -200 MPa), thus acting as a barrier for the interfacial dislocation movement. Therefore the dislocation can not overcome this stress barrier if the applied stress is low. It stops and aligns parallel to the γ' -edge, i.e. it is transformed from a 60° - dislocation into an edge dislocation. Such interfacial edge dislocation with $\langle 001 \rangle$ orientation are typical for networks in modern nickel-base superalloys, e.g. TMS138, where the γ/γ' -misfit is high and therefore the effects of misfit stresses pronounced.

5. Conclusions

1. It is shown experimentally that under low applied stresses the matrix dislocations frequently cross glide with the periodicity length equal to that of the γ/γ' -microstructure. The reason for this effect are inhomogeneous misfit stresses, which control the dislocation movement in such a way, that the resulting interfacial dislocation segments relax most effectively the misfit stresses. An outstanding role in this frequent cross glide is played by the shear components of the misfit stress. They are low on average but near the γ' -edges attain a high value. The result is, that the dislocation loops follow an average $\langle 001 \rangle$ direction on a zigzag course, being reflected when approaching R-channels parallel to this $\langle 001 \rangle$ -direction, and cross slipping when traversing C-channels perpendicular to this direction. Straightening of the interfacial zigzag dislocation into $\langle 001 \rangle$ oriented edge dislocations is an additional effect of the shear stresses. Two sets of these $\langle 001 \rangle$ dislocations can react, forming $\langle 001 \rangle$ oriented square networks.
2. The described effects occur when the magnitude of misfit stresses is not much lower than the magnitude applied stress. This is the case of annealing or creep under low applied stress.

3. The effects also should increase with temperature due to the increase of the absolute value of γ/γ' -misfit.
4. In single-crystal nickel-base superalloys of the newest generations with a high concentration of refractory elements the γ/γ' -misfit is very high. The described effects explain the formation of very regular square networks with $\langle 001 \rangle$ orientation as observed, e.g. in TMS138.
3. Cross glide at C-channels was explained only qualitatively and needs further investigation. For detailed understanding of its mechanism modeling of this process by dislocation dynamics is required.

Acknowledgements

Authors thank the German Research Foundation (DFG) for funding of this work (project PO 405/10-2, LI 494/4-2) and Mr. D. Noack (BAM, Berlin) for assistance in finite element modelling.

References

[1] L.J. Carroll, Q. Fang and T.M. Pollock, Metall. Trans. A 39A (2008) p.1290.

[2] A. Sato, A.Ch. Yeh, T. Kobayashi, T. Yokokawa, H. Harada, T. Murakumo and J. Zhang, *A 5th generation Ni-base single crystal superalloy with superior elevated temperature properties*, in *Materials for advanced power engineering*, J. Lecomte-Beckers, M. Carton, F. Schubert and P.J. Ennis, eds., Forschungszentrum Jülich GmbH, Jülich, 2006, p.287.

[3] J.X. Zhang, J.C. Wang, H. Harada, and Y. Koizumi, Acta Mater. 53 (2005) p.4623.

[4] J.X. Zhang, Y. Koizumi, T. Kobayashi, T. Murakumo and H. Harada, Metall. Trans A 35A (2004) p.1911.

[5] T.M. Pollock and A.S. Argon, Acta Metall. and Mater. 42 (1994) p.1859.

[6] W. Schneider, *Hochtemperaturkriechverhalten und Mikrostruktur der einkristallinen Nickelbasis-Superlegierung CMSX-4 bei Temperaturen von 800°C bis 1100°C*, University of Erlangen-Nürnberg, Erlangen, 1993.

[7] M. Feller-Kniepmeier and T. Link, Mat. Sci. and Eng. A 113 (1989) p.191.

[8] A.K. Singh, N. Louat and K. Sadananda, Metall. Trans A 19A (1988) p.2965.

[9] A. Fredholm and J.L. Strudel, *High temperature creep mechanisms in single crystals of some high performance nickel base superalloys*, in *High Temperature Alloys, their Exploitable Potential*, J.B. Mariott, H. Herz, J. Nihoul and J. Ward, eds., Elsevier Applied Science, London, 1987, p.9.

[10] U. Brückner, A. Epishin and T. Link, Acta Mater. 45 (1997) p.5223.

[11] A. Epishin, T. Link and U. Brückner, Influence of dendritic inhomogeneity on creep behaviour of a single-crystal superalloy of the 3rd generation, DFG project PO 405/4-1; LI 494/3-1, interim report, BAM-TU Berlin, 2002.

[12] U. Brückner, BAM Berlin, unpublished research, 2008.

[13] U. Glatzel, *Microstructure and Internal Strains of Undeformed and Creep Deformed Samples of a Nickel-Base Superalloy*, Verlag Dr. Köster, Berlin, 1994.

[14] A. Epishin, T. Link and G. Nolze, J. of Microscopy 228 (2007) p.110.

- [15] M. Fährmann, W. Hermann, E. Fährmann, A. Boegli, T.M. Pollock and H.G. Sockel, Mat. Sci. and Eng. A260 (1999) p.212.
- [16] D. Siebörger, H. Knake and U. Glatzel, Mat. Sci. and Eng. A298 (2001) p.26.
- [17] B. Fedelich, J. Beckmann, M. Finn, M. Zelewski and J. Olschewski, Determination of temperature dependent elastic constants of anisotropic materials by the resonance method, Materials week 2002, DGM.e.V., 30.9-2.10.2002, Munich (CD-Rom).
- [18] J.A. Nairn, Mechanics of Materials 26 (1997) p.63.
- [19] T. Link, A. Epishin, M. Klaus, U. Brückner and A. Reznicek, Mat. Sci. and Eng. A 405 (2005) p.254.
- [20] B.A Bilby, R. Bullough and E. Smith, Proc. Roy. Soc. A231 (1995) p.263.

Appendix: Glide force under resolved shear stress

The projection of the Peach-Koehler formula force into the glide plane and perpendicular to the dislocation line vector $\vec{\xi}$ gives the glide component of the elastic force acting on a dislocation:

$$\mathbf{F}^{glide} = \left\{ \left[\left(\hat{\sigma} \cdot \vec{b} \right) \times \vec{\xi} \right] \cdot \left(\vec{n} \times \vec{\xi} \right) \right\} \quad (1A)$$

where $\hat{\sigma}$, $\vec{b}$, $\vec{\xi}$ and $\vec{n}$ are respectively the stress tensor, the Burgers and line vectors, and the normal of the glide plane. Use of the Lagrange identity $(\vec{A} \times \vec{B}) \cdot (\vec{C} \times \vec{D}) = (\vec{A} \cdot \vec{C})(\vec{B} \cdot \vec{D}) - (\vec{A} \cdot \vec{D})(\vec{B} \cdot \vec{C})$ with $A = \hat{\sigma} \cdot \vec{b}$, $B = \vec{\xi}$, $C = \vec{n}$ and $D = \vec{\xi}$ gives:

$$\vec{F}^{glide} = \left[\left(\hat{\sigma} \cdot \vec{b} \right) \cdot \vec{n} \right] \cdot \left(\vec{n} \times \vec{\xi} \right) \quad (2A)$$

$$\mathbf{F}^{glide} = \left(\hat{\sigma} \cdot \vec{b} \right) \cdot \vec{n} \quad (2B)$$

Table 1. Investigated single crystal superalloys: Composition in wt%. Constrained misfit, measured at RT in the primary dendrite arm (PDA).

Alloy Company	Al	Ti	Cr	Co	Ni	Mo	Ru	Ta	W	Re	Hf	Constrained misfit, % Reference
SRR99 RR,UK	5.5	2.2	8.5	5.0	66.5	-	-	2.8	9.5	-	-	≈-0.2 [10]
CMSX-4 C&M, USA	5.6	1.0	6.5	10.0	61.3	0.6	-	6.0	6.0	3.0	-	≈-0.4 [10]
CMSX-10 C&M, USA	5.6	0.23	2.3	3.3	68.1	0.4	-	8.3	5.5	6.3	-	≈-0.1 [11]
TMS138 NIMS, Japan.	5.9	-	2.9	5.9	63.9	2.9	2.0	5.6	5.9	4.9	0.1	≈-1.0 [12]

1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
20
21
22
23
24
25
26
27
28
29
30
31
32
33
34
35
36
37
38
39
40
41
42
43
44
45
46
47
48
49
50
51
52
53
54
55
56
57
58
59
60

Table 2. Creep and annealing tests in the present investigation.

Material	T, °C	σ , MPa	t, h	ϵ , %	Fig.
CMSX-4	850	650	0.4	0.35	1
CMSX-4	1100	120	4	0.1	-
CMSX-4	1100	120	25	0.3	-
CMSX-10	1100	120	25	0.12	2a
TMS-138	1100	137	2	0.04	3
SRR99	980	200	45.4	0.3	4
SRR99	980	200	5	0.06	5
CMSX-4	980	0	1000	0	2b
SRR99	1100	0	300	0	6a, b
CMSX-10	1050	0	500	0	-

Table 3. Misfit networks with square or rectangular $\langle 100 \rangle$ -meshes and interfacial dislocations with $\langle 100 \rangle$ line direction observed by different groups.

Observation	Material	Test parameters	Reference, Fig.
$\langle 100 \rangle$ -meshes rectangular	MK-4	annealing 1050°C, 1150 h	[1], Fig. 5b
$\langle 100 \rangle$ -meshes square	TMS-196	creep 1000°C, 245 MPa	[2], Fig. 3b
$\langle 100 \rangle$ -meshes square	TMS-138	creep 1100°C, 245 MPa, 20 h or until rupture	[3], Fig. 5b, 6b, 7b
$\langle 100 \rangle$ -meshes square	TMS-162	creep 1100°C, 137 MPa, rupture	[4], Fig. 5d
$\langle 100 \rangle$ interfacial dislocations	CMSX-3	creep 1050°C, 50 MPa, 48 h	[5], Fig. 9
$\langle 100 \rangle$ interfacial dislocations	CMSX-4	annealing 950°C, 3655 h	[6], Fig. 23a
$\langle 100 \rangle$ -meshes rectangular	CMSX-4	annealing 1100°C, 224 h	[6], Fig. 23b
$\langle 100 \rangle$ -meshes square	CMSX-4	creep 1100°C, 140 MPa, 0.11%	[6], Fig. 85a
$\langle 100 \rangle$ interfacial dislocations	SRR99	annealing 980°C, 1000 h	[7], Fig. 5
$\langle 100 \rangle$ interfacial dislocations	IN713c	annealing 1000°C, 8 h	[8], Fig. 5a, b
$\langle 100 \rangle$ interfacial dislocations	alloy 221	creep 1050°C, 140 MPa, 1 h	[9], Fig. 5b

Figure captions:

Figure 1. Interfacial dislocations in CMSX-4, creep deformed at 850°C, 650 MPa, 0.4 h, 0.35%.

Two channels marked by arrows. Cross section, weak beam, beam direction $\vec{k}_0 = [001]$, diffraction vector $\vec{g} = 0\bar{6}0$. Vertical line in the middle results from the montage of two images.

Figure 2. a) Interfacial dislocations in CMSX-10, creep deformed at 1100°C, 120 MPa, 25 h, 0.12%.

Cross section, bright field (BF), $\vec{k}_0 = [100]$, $\vec{g} = 002$.

b) In CMSX-4, annealed at 980°C for 1000 h. BF, $\vec{k}_0 = [001]$, $\vec{g} = 020$.

Figure 3. Dislocation loop expanding in a matrix channel during primary creep. TMS-138 1100°C, 137 MPa, 2 h, 0.04%. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 020$. Tilted line in the middle results from the montage of two images.

Figure 4. Interfacial dislocations with $\vec{b} = a/2[101]$, propagating in $[010]$ average direction. Cross slip takes place directly on the edge of C-channels. TMS 138, 1100°C, 137 MPa, 2 h, 0.04%, cross section. BF, $\vec{k}_0 = [100]$, $\vec{g} = 020$.

Figure 5. Dislocation loops in SRR99 creep deformed at 980°C, 200 MPa, 45.4 h, 0.3%.

a) Viewing direction perpendicular onto the (001) interface. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 0\bar{2}0$.

b) Foil tilted with (111) glide plane edge on. BF, $\vec{k}_0 = [\bar{1}01]$, $\vec{g} = 1\bar{1}1$.

Figure 6. Square interfacial networks in SRR99, annealed at 1100°C for 300 h. (001) cut, imaged either by TEM in BF, multibeam (a) or by SEM (b).

Figure 7. Dislocation loops align in $\langle 100 \rangle$ directions and form square networks. TMS138, 1100°C, 137 MPa, 2 h, 0.04%. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 220$. Tilted line in the middle results from the montage of two images.

Figure 8. Structural model for FEM calculations, given in a (100) projection. The analysed area is cubic and extends from the center of a γ' cube to the center of the crossing matrix channels.

Figure 9. Distributions of misfit stresses in MPa in (001) sections of the P-channel at different distances from the interface: components σ_{22}^m (a) and σ_{33}^m (b) at the distance $0.5w$, components σ_{13}^m (c) and σ_{12}^m (d) at the distance $0.03w$. The positions of the sections are shown in Fig. 8.

Figure 10. Kink in a dislocation loop. The loop moves in a P-channel lying between two γ' cubes.

Figure 11. Distributions of RSS $\tau_{(111)}^m$ in MPa in 3 cross sections along the (001) -channel: in the channel middle $0.5w$ (a), $0.25w$ from the (001) interface (b) and very close to the interface $0.03w$ (c).

Figure 12. Resolved shear stresses $RSS(x, y, z)$ acting on dislocations gliding in the P-channel (001) .

a) $\tau_{(111)}^m = \tau_{(\bar{1}\bar{1}1)}^m$ for $x = 0$ in y-direction across the R-channel (010) .

b) $\tau_{(111)}^m$ for $y = 0$ in x-direction across the C-channel (100) . RSS is plotted for 3 distances from the γ/γ' -surface.

The coordinates $x = 0$, $y = 0$ belong to the corner bottom-left in Fig. 11c.

Formatted: Bullets and Numbering

Figure 13. Cross glide when crossing the C-channel (100).

Figure 14. Distribution of $\tau_{(111)}^m$ in MPa on a longitudinal (010) section with $y=0$. (Middle of the cube edge in [010] direction).

Figure 15. Distribution of $F_{[010]}(\hat{\sigma}^m)$ in MPa on a cross section for $\sigma_{33}^a = 0$. $F_{[010]}$ drives the dislocation segment with $\vec{b} = \frac{a}{2}[0\bar{1}1]$ and $\vec{\xi} = [100]$ towards the [100] edge. ($|\vec{b}|$ is taken to be equal 1).

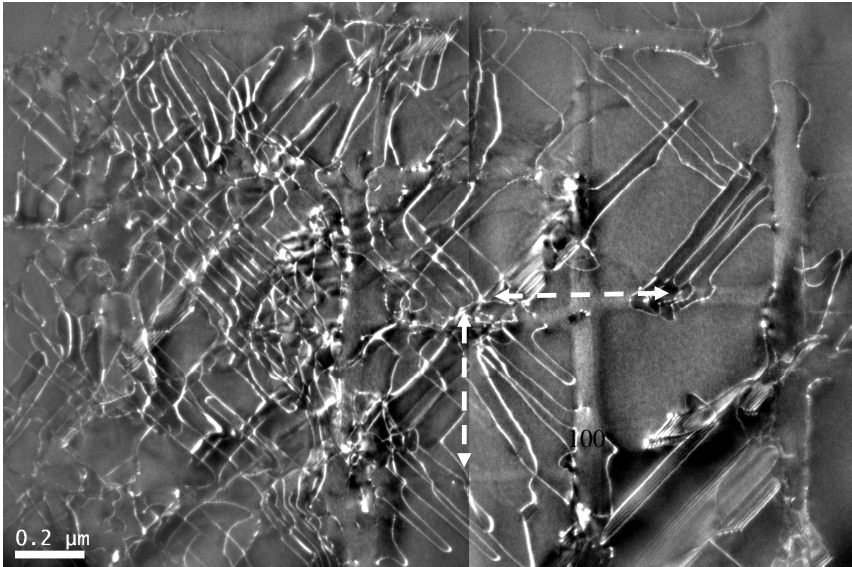


Figure 1. Interfacial dislocations in CMSX-4, creep deformed at 850°C, 650 MPa, 0.4 h, 0.35%. Two channels marked by arrows. Cross section, weak beam $\vec{k}_0 = [001]$, $\vec{g} = 0\bar{6}0$. Vertical line in the middle results from the montage of two images.

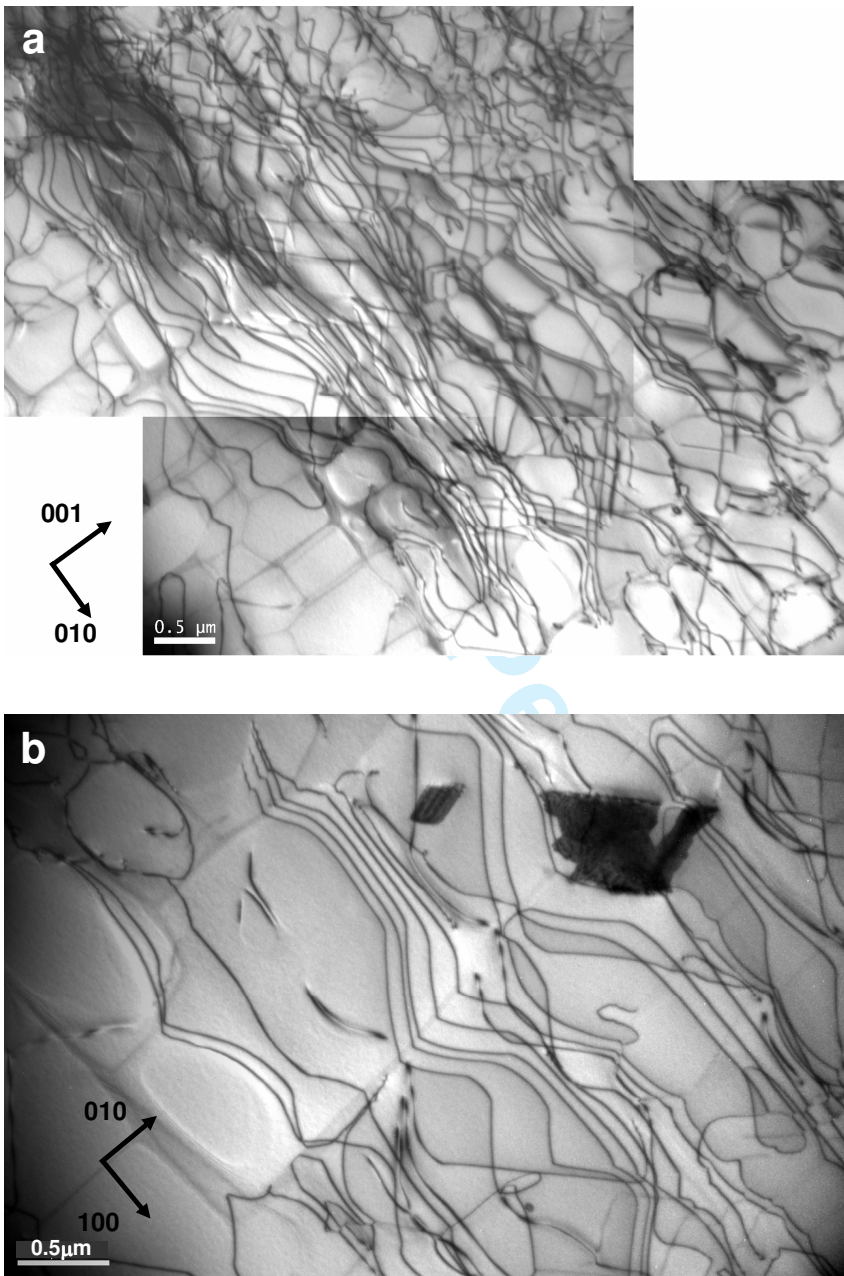


Figure 2.

a) Interfacial dislocations in CMSX-10, creep deformed at 1100°C, 120 MPa, 25 h, 0.12%.

Cross section, bright field (BF), beam direction $\vec{k}_0 = [100]$, diffraction vector $\vec{g} = 002$.

b) In CMSX-4, annealed at 980°C for 1000 h. BF, $\vec{k}_0 = [001]$, $\vec{g} = 020$.

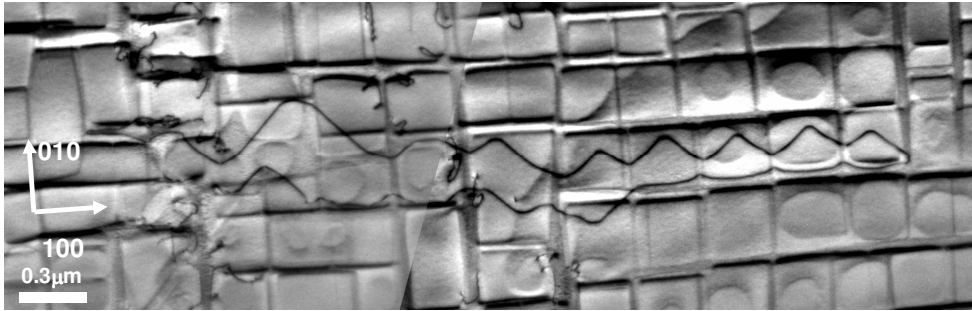


Figure 3. Dislocation loop expanding in a matrix channel during primary creep. TMS-138 1100°C, 137 MPa, 2 h, 0.04%. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 020$. Tilted line in the middle results from the montage of two images.

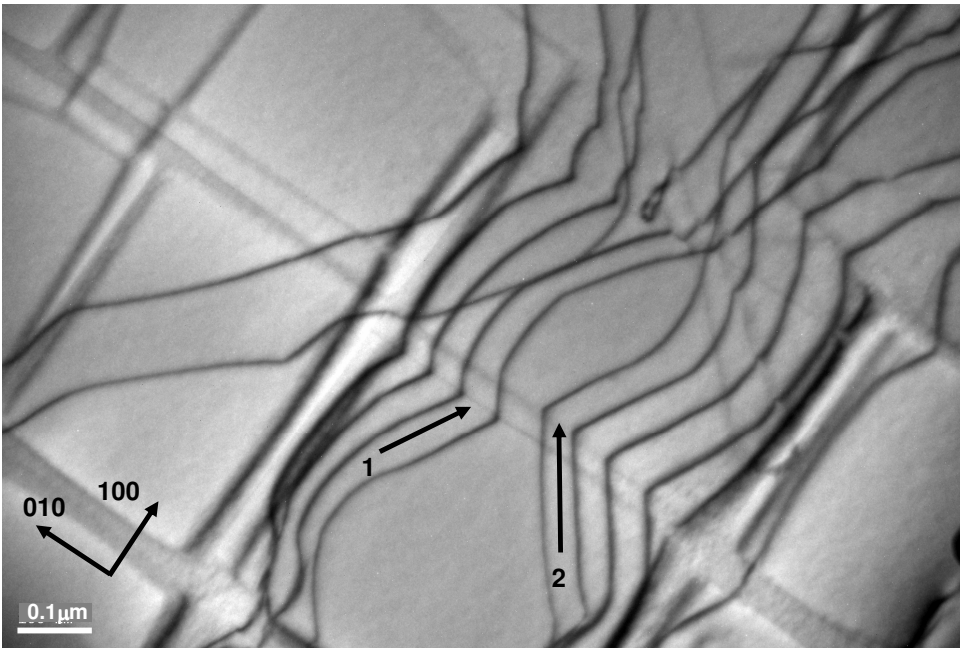


Figure 4. Interfacial dislocations with $\vec{b} = a/2[101]$, propagating in $[010]$ average direction. Cross slip takes place directly on the edge of C-channels. TMS 138, 1100°C, 137 MPa, 2 h, 0.04%, cross section. BF, $\vec{k}_0 = [100]$, $\vec{g} = 020$.

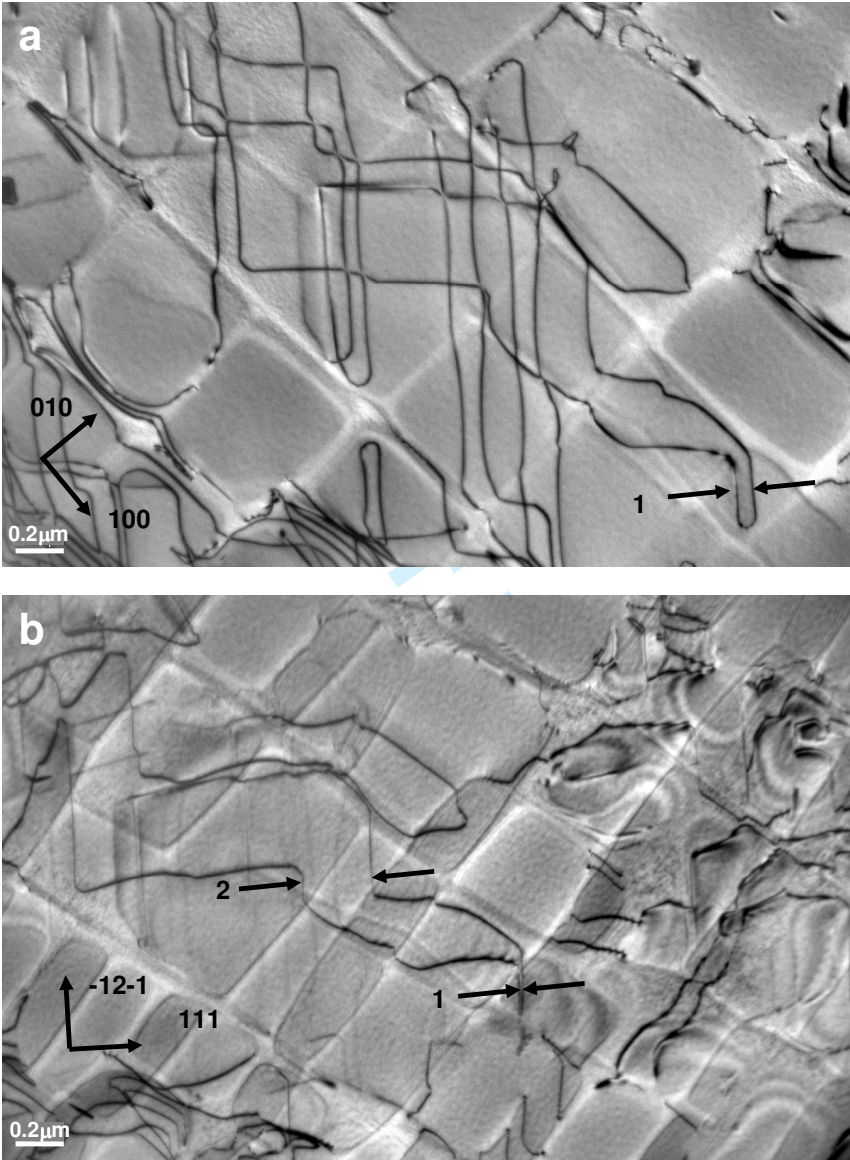


Figure 5. Dislocation loops in SRR99 creep deformed at 980°C, 200 MPa, 45.4 h, 0.3%.
a) Viewing direction perpendicular onto the (001) interface. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 0\bar{2}0$.
b) Foil tilted with (111) glide plane edge on. BF, $\vec{k}_0 = [\bar{1}01]$, $\vec{g} = 1\bar{1}1$.

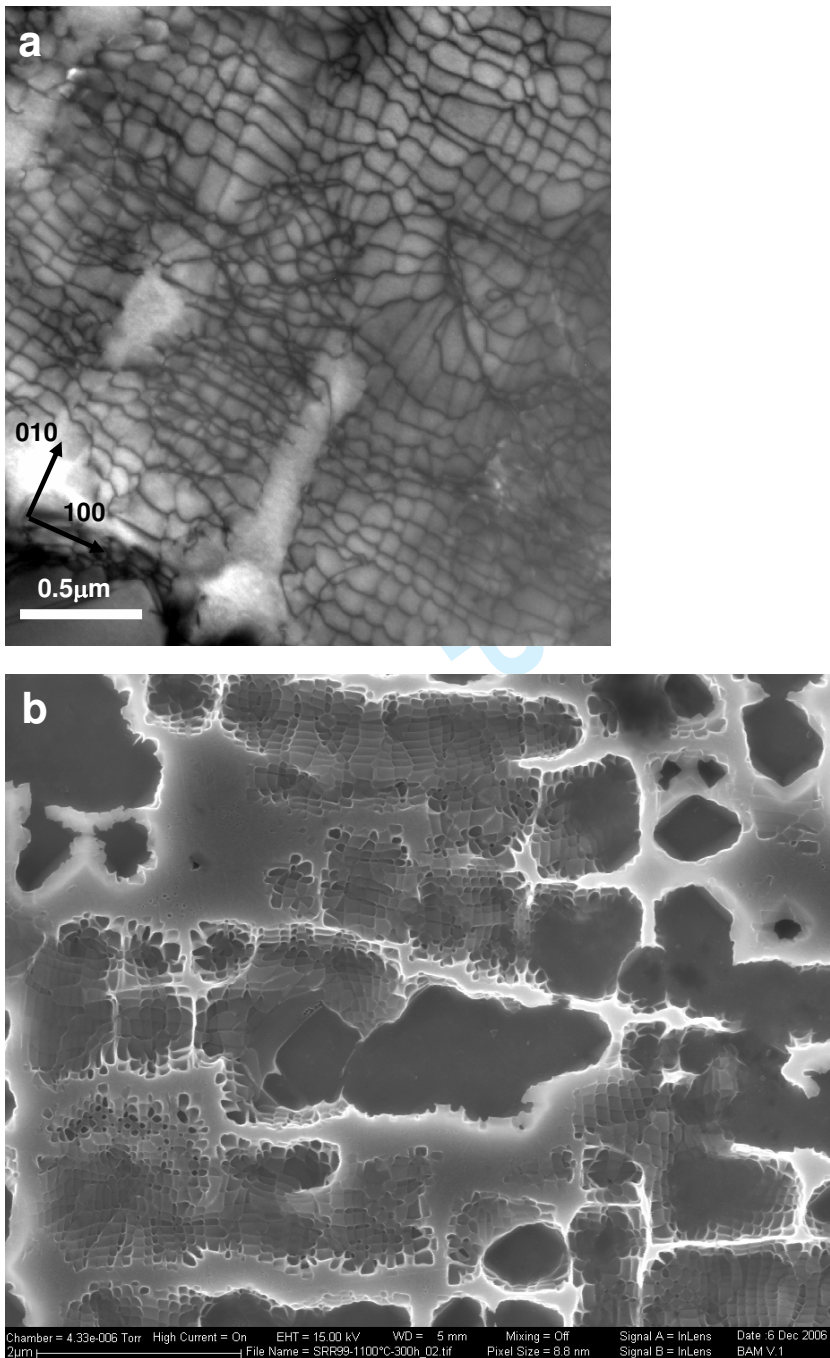


Figure 6. Square interfacial networks in SRR99, annealed at 1100°C for 300 h. (001) cut, imaged either by TEM in BF, multibeam (a) or by SEM (b).

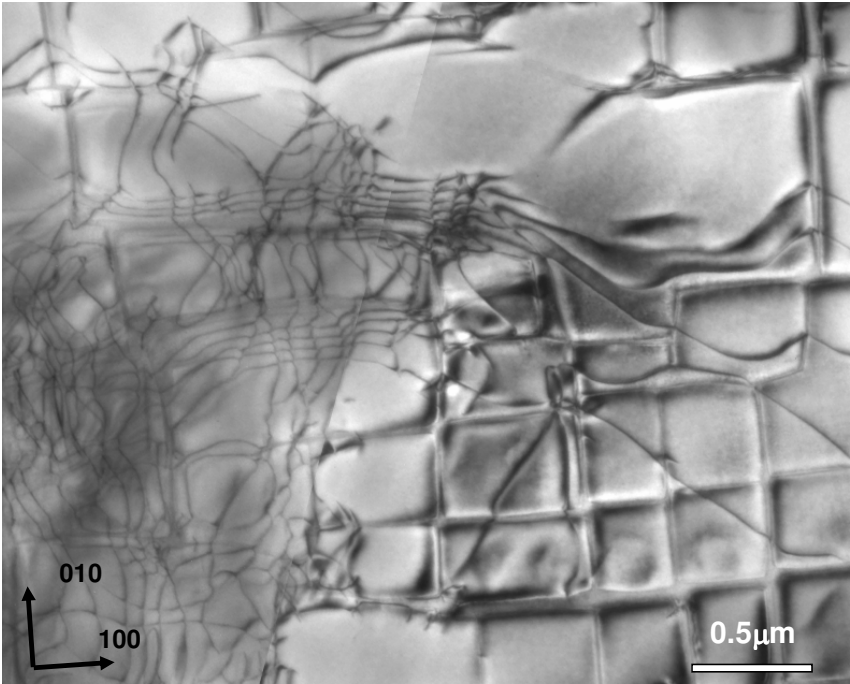


Figure 7. Dislocation loops align in $\langle 100 \rangle$ directions and form square networks. TMS138, 1100°C, 137 MPa, 2 h, 0.04%. Cross section, BF, $\vec{k}_0 = [001]$, $\vec{g} = 220$. Tilted line in the middle results from the montage of two images.

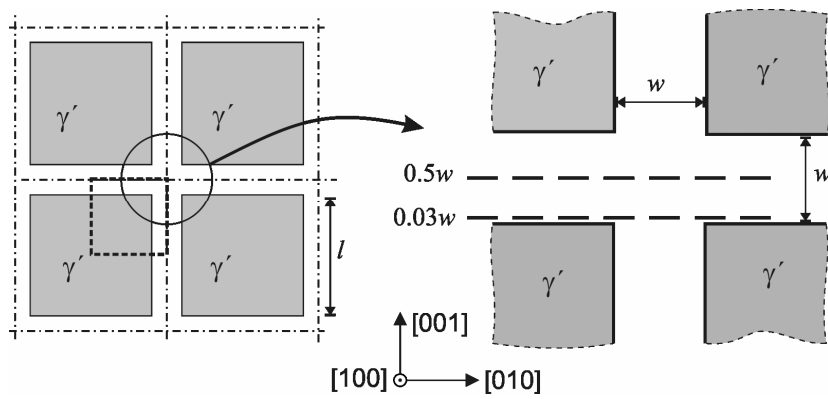


Figure 8. Structural model for FEM calculations, given in a (100) projection. The analysed area is cubic and extends from the center of a γ' cube to the center of the crossing matrix channels.

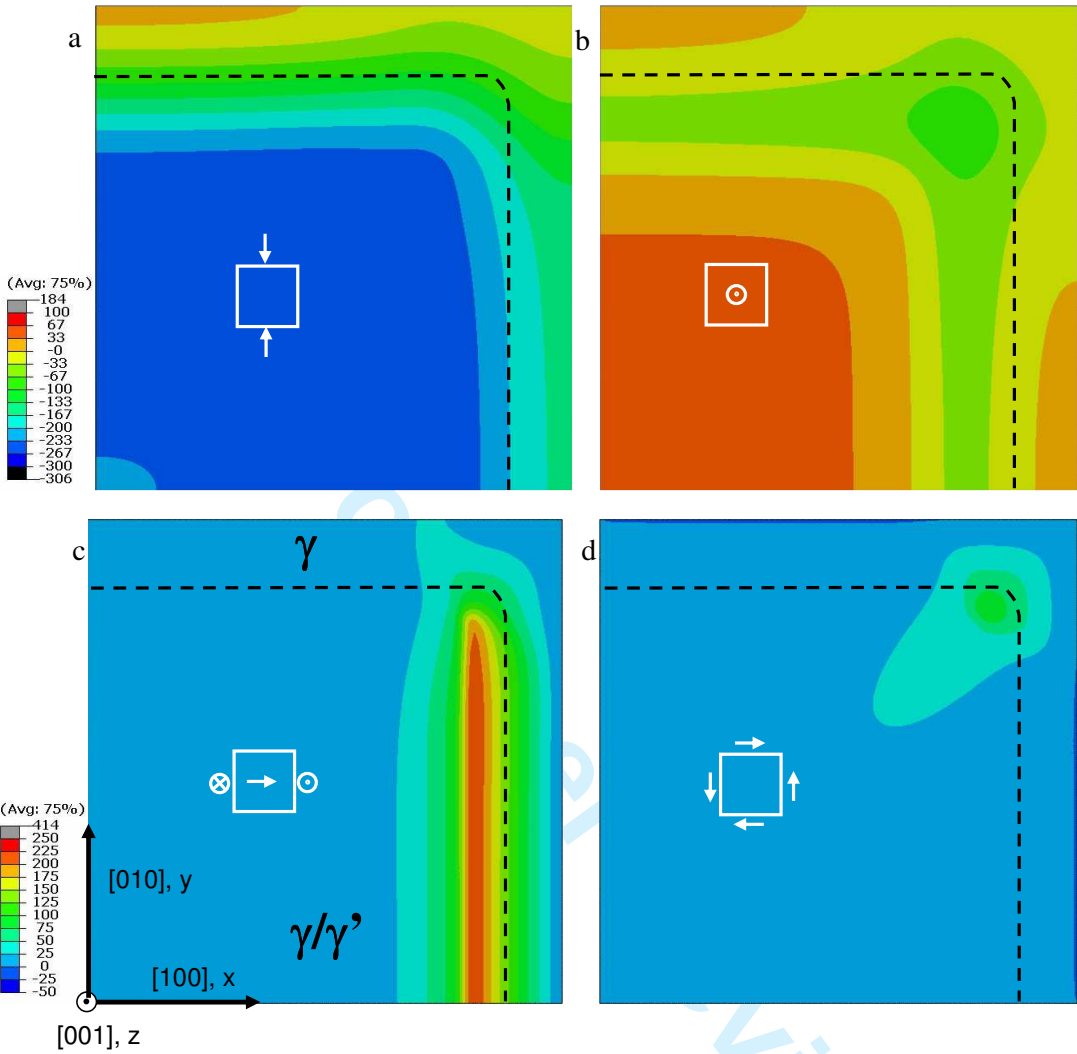


Figure 9. Distributions of misfit stresses in MPa in (001) sections of the P-channel at different distances from the interface: components σ_{22}^m (a) and σ_{33}^m (b) at the distance $0.5w$, components σ_{13}^m (c) and σ_{12}^m (d) at the distance $0.03w$. The positions of the sections are shown in Fig. 8.

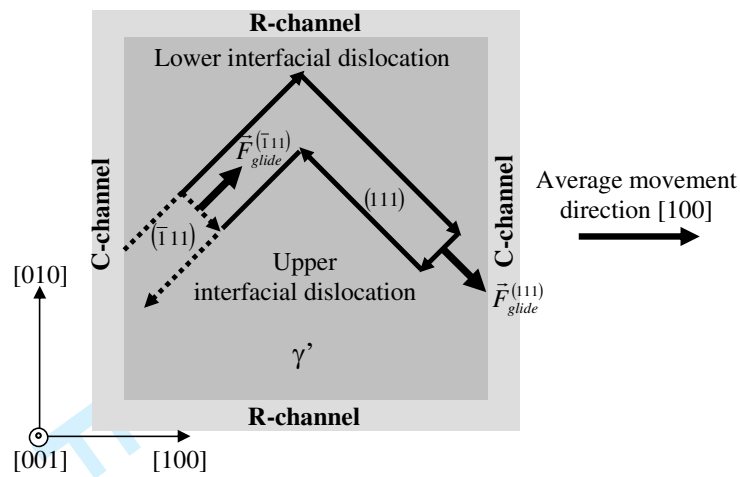


Figure 10. Kink in a dislocation loop. The loop moves in a P-channel lying between two γ' cubes.

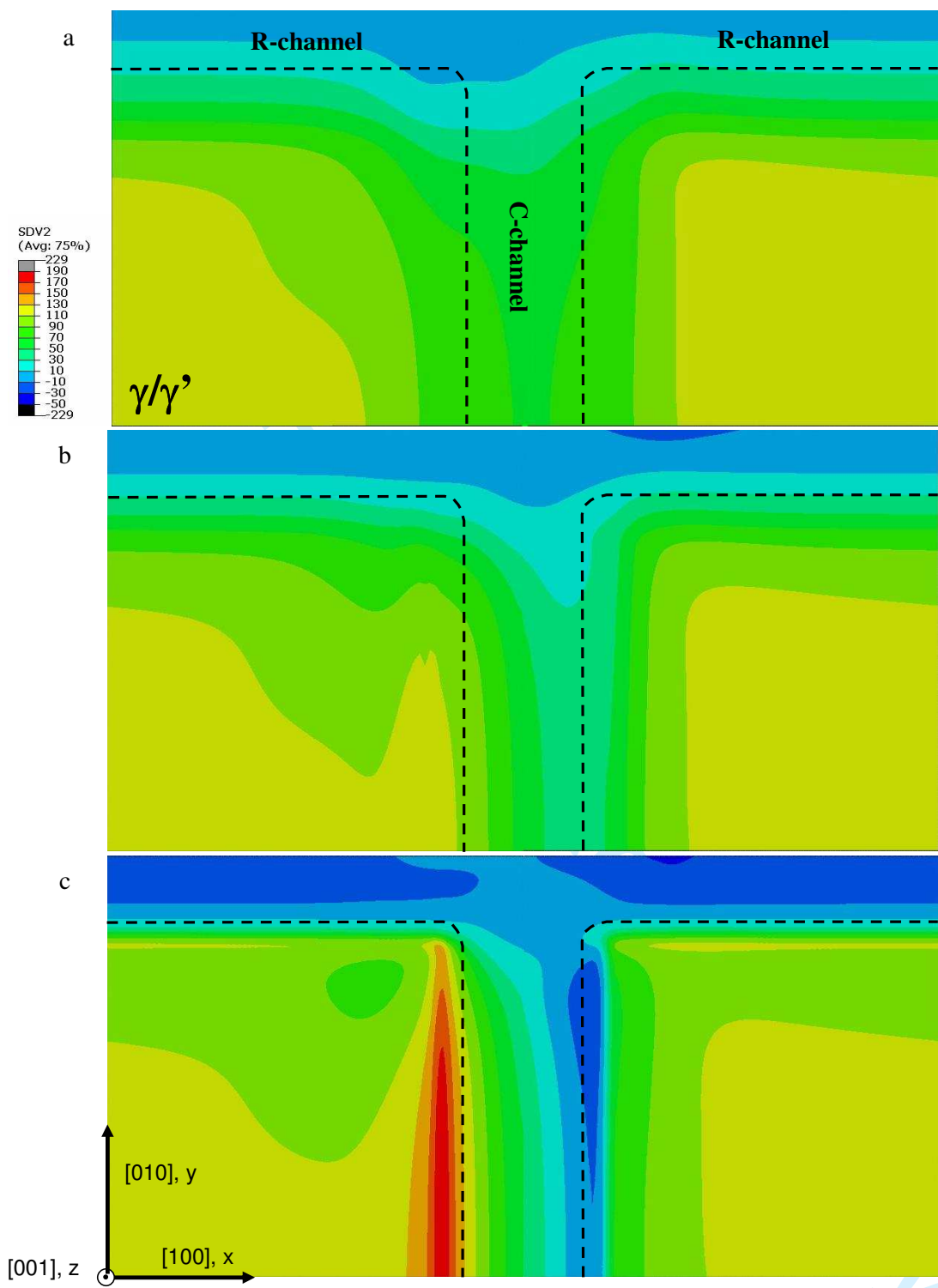


Figure 11. Distributions of RSS $\tau'''_{(111)}$ in MPa in 3 cross sections along the (001)-channel: in the channel middle $0.5 w$ (a), $0.25 w$ from the (001) interface (b) and very close to the interface $0.03 w$ (c).

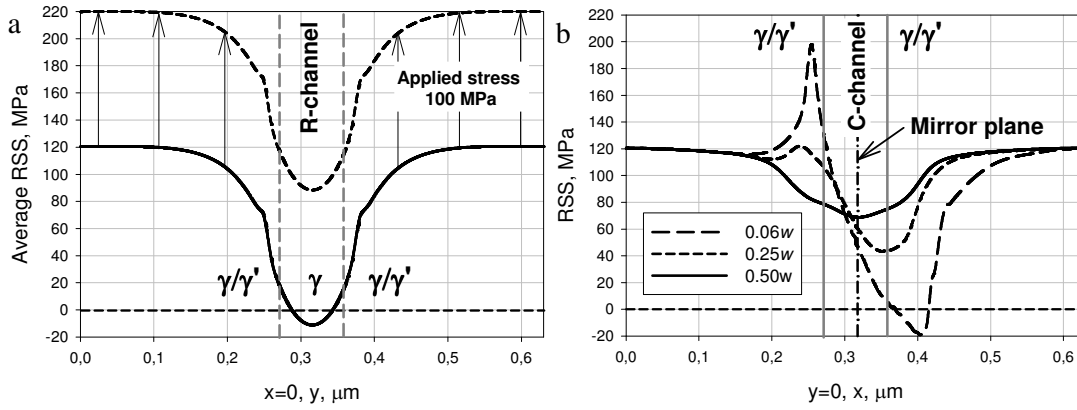


Figure 12. Resolved shear stresses $RSS(x, y, z)$ acting on dislocations gliding in the P-channel (001).

a) $\tau_{(111)}^m = \tau_{(\bar{1}\bar{1}\bar{1})}^m$ for $x = 0$ in y -direction across the R-channel (010).

b) $\tau_{(111)}^m$ for $y = 0$ in x -direction across the C-channel (100). RSS is plotted for 3 distances from the γ/γ' -surface.

The coordinates $x = 0, y = 0$ belong to the corner bottom-left in Fig. 11c.

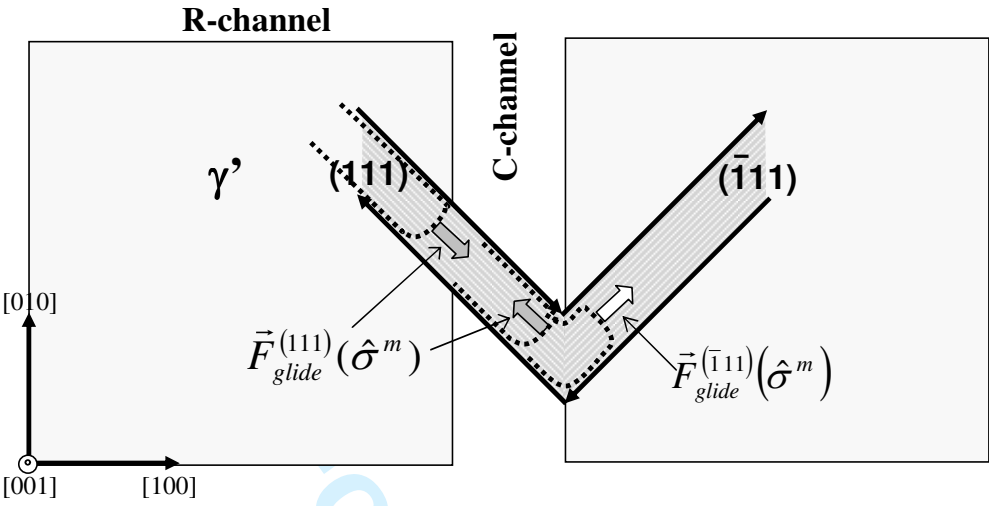


Figure 13. Cross glide when crossing the C-channel (100).

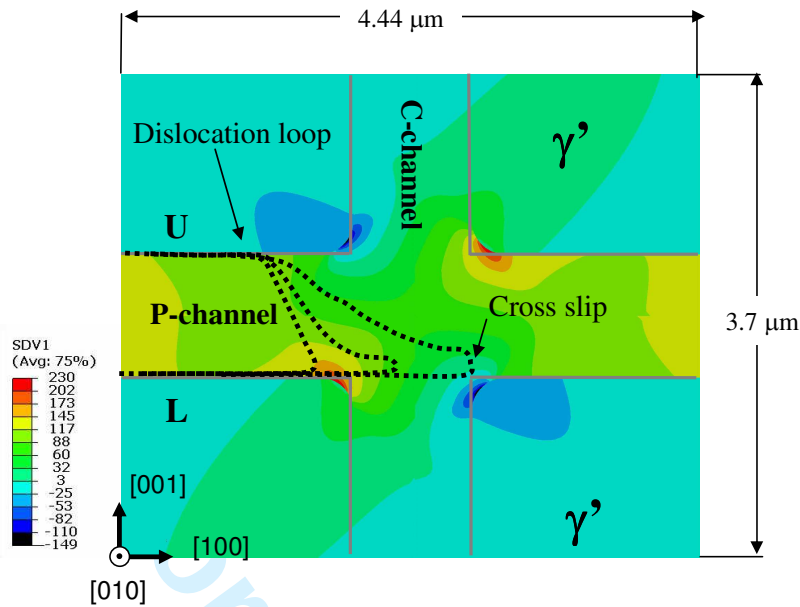


Figure 14. Distribution of $\tau'''_{(111)}$ in MPa on a longitudinal (010) section with $y=0$. (Middle of the cube edge in [010] direction).

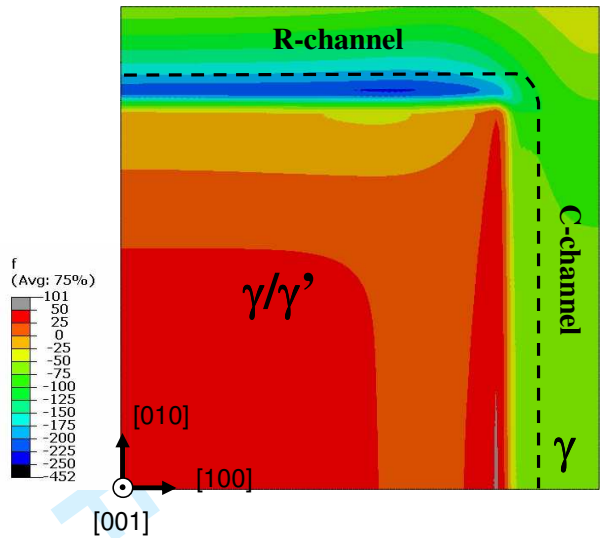


Figure 15. Distribution of $F_{[010]}(\hat{\sigma}^m)$ in MPa on a cross section for $\sigma_{33}^a = 0$. $F_{[010]}$ drives the dislocation segment with $\vec{b} = \frac{a}{2}[0\bar{1}1]$ and $\vec{\xi} = [100]$ towards the [100] edge. ($|\vec{b}|$ is taken to be equal 1).