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Numerical Investigation of In Situ TEM Tensile Tests

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The microstructure and material damage due to elastic-plastic strains can be investigated by means of tensile tests in the transmission electron microscope. Owing to the non-standard specimen geometries which are adapted to the time-consuming preparation methods, it is only possible to make conditional statements about the stresses and strains operating in the region of observation. For this reason, a numerically based method was developed to determine a correlation between the displacement of the specimen's loading-fixture and the locally operating stresses or strains observed in the material region. Here, the TEM specimen geometry is firstly modelled using FE-software. Following this, a tensile test is numerically simulated for this geometry by means of the FE-software and the corresponding determined stresses or strains are ascertained for the respective displacements. In this way, it is possible to correlate the microstructural changes observed in the transmission electron microscope with the local strains during the in situ tests.

Keywords: IN SITU TENSILE TESTS, TEM, FEM

Introduction

During the innovative method of sheet-bulk deformation, functional components are manufactured directly from thin sheet [1]. The complex deformation processes possessing high deformation levels can lead to the development of ductile damage in the material which limits the material's residual load carrying capacity [2]. Depending on the level of deformation, pores form in the material which coalesce during continuing deformation and develop into cracks. However, a verification of earlier stages of this pore formation, which is necessary to successfully design a process, is only possible at high magnifications using transmission electron microscopy. The material's ductile damage has already been the main focus of deformation simulations. It is fundamental for the accuracy of the computed results to specify the pore-volume as early as possible after their occurrence during the manufacturing process. Hitherto, the initial pore-volume had been defined using chemical analyses, which relies on the total volume of precipitates. An analysis of the actual process of pore development by means of in situ observations in the transmission electron microscope with the aid of a straining holder is a promising improvement. As soon as the first cavities form, this development can be verified. In addition to this, the grain's crystallographic orientation can be determined via diffraction patterns. Information about the level of deformation, pore-volume and grain orientation thus enables the modelling of damage development to be improved.

State of the art

Investigations of metals' and metallic alloys' microstructures at the nano-scale has gained more importance over the last few years [3]. On the one hand, this is because of the growing use of microelectronic and microelectronic-mechanical systems in different fields of electronics, automation or mechanical and instrument engineering. These systems possess a range of new and previously unexplored properties which play a decisive role in complex systems. As a rule, the entire system depends on the precision and stability of its functional capability. On the other hand, nano-scale tests on specimens using various types of loading permit fundamental knowledge in the field of metal structures to be extended. The understanding of fundamental mechanisms, which form the basis of plastic and elastic deformation, can be provided in this way. In addition to this, the scaleability limits of macroscopically measured material parameters can be determined for applications at the nano-scale.

Numerous investigations; see for example [4, 5], comprise of both aspects of materials science and production technology of micro-systems mentioned above. Metallic coatings having thicknesses of approx. 100 nm are frequently employed in the latter field. For this reason, experimental testing equipment based on micro systems, which consist of such structural elements, are described in many publications [6]. The most widely employed production process for manufacturing thin metallic specimens is deposition of metal onto the surface of another material using subsequent local ablation, or sublimation and condensation onto a glass surface with subsequent specimen ablation [7]. The specimens are manufactured from aluminium, silicon, copper, titanium, nickel [8] and other materials. By means of such procedures, residual stresses in the material are avoided. These stresses occur during all mechanical processing and can decisively influence the investigation's results for such small specimens.

Tension, compression and bending [9, 10] are the most important modes of loading for this type of material testing. The material specimens are frequently subjected to tensile tests. The tensile loading enables a complete analysis to be carried out of the elastic, plastic and strength properties including analyses at the dislocation level. For this reason, the specimens are mainly subjected to uniaxial tensile loading. The difficulty here is that it is necessary to employ special testing equipment for introducing low forces; in the range smaller than μN, over very short displacements. The test results are directly dependent on the precision of the equipment [7]. Besides this, an important criterium for the test procedure is that the force must be applied exactly parallel to the specimen's axis. This provides a uniform stress distribution over the specimen's cross-section. In the case of non-parallelity, the measurement error can be as much as 150 %, even for very small angles [3].

For the tensile test, the force and change in specimen length are measured as initial data with the help of suitable measuring instruments. From this data, the stress can be computed as a function of the specimen strain. Thus by means of the in situ tensile tests, the mechanical properties can be determined based on the primary parameters; extension and stress.

In previous investigations, it was established that it is necessary to take into account the change in the deformation mechanism during the transition from macro-specimens to micro and nano-specimens [3]. This change is associated with an unusual behaviour of the dislocations:

Thus for example, dislocation channeling [11] in thin metal films or dislocation starvation [12] or dislocation nucleation/escape [13] is observed in sub micron single crystals. Furthermore, a reduction in the microstructure's size can lead to unusual properties, such as plastic strain recovery, irrespective of specimen [14, 15].

Therefore, a reduction in the dislocation density can be observed in a thin specimen. The dislocations also exit the specimen without external loading even prior to the commencement of deformation. For this reason, further plastic deformation of the specimen requires renewed dislocation formation. This elevates the metal's yield stress. It was established [7] that the yield stress value of approx. 100 nm thin aluminium specimens is increased by a factor of up to 60 times that for common size specimens. Here, the Young's modulus remains almost unchanged. Besides this, a Bauschinger effect is observed to occur extraordinarily early. This effect is caused by large grains plastically and not elastically deforming for an identical loading. It was established that the deformation occurred along the grain boundaries. The deformation initially occurs at the grain boundaries which lie at an angle of 45° to the axis of the force application. This is caused by the maximum stresses which occur here. Corresponding to this, crack formation is initiated at an angle of 45° to the specimen's edge prior to the crack aligning normal to the force application axis during further deformation. Mathematical models, using finite element methods, are frequently implemented in order to enable more accurate data to be obtained and a deeper analysis of such processes to be performed. The developed models can usually be divided into two groups. The modelling of the elastic deformation of the specimen holder or the individual elements during the introduction of the force [4, 16] and the modelling of the grains' behaviour; that is, the deformation and orientation change of the individual crystallites [8, 17-19].

Objectives

To assess the microstructure's development and material damage of deep-drawing steel during the bulk forming of sheet, it is necessary to carry out TEM analyses which identify incipient pore formation. Although these types of tests can be performed in the TEM by means of in situ tensile tests, it has hitherto only been possible to partially correlate observed microstructural changes with the locally occurring elastic-plastic strains.

The deposition specimen-manufacturing procedure mentioned above is unsuitable in the present case since tests have to be preformed using specimen materials of engineering alloys from actual components. For this reason, suitable tensile specimens can not be produced for in situ TEM analyses based on usual specimen geometries.

Instead of this, a specimen preparation methodology must be resorted to in which the specimens are thinned by means of FIB milling, although this can lead to a certain input of gallium ions, the formation of an amorphous surface layer and to a surface roughening [3].

To be able to attribute the microstructural behaviour observed in the tensile test to the specified strain or stress state, the essential aim of this work is to correlate the displacement of the straining holder with the local strains in the observed region of the TEM specimen. Here, it should be taken into account that depending on the preparation, the TEM tensile specimens used exhibit a non-uniform thickness over their cross-sections.

For this reason, the stresses and deformations can not be directly determined by means of analytical conversions. Accordingly, the mathematical modelling of the TEM tensile tests accompanies the in situ experimental investigations by taking into account the specimens geometric shape. Employing the numerical 3D-finite element model, local stresses and deformations are computed from the externally applied forces exerted on the specimen as a whole for the entire specimen geometry.

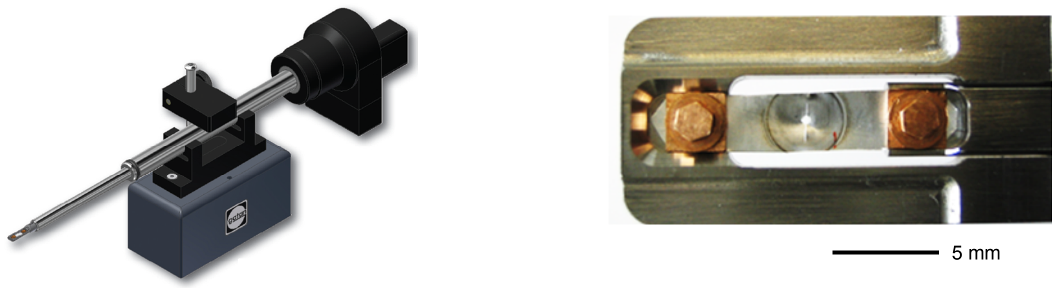
Specimen preparation

In order to obtain the tensile specimen's electron-transparency which is sufficient for observations using TEM, the specimen preparation routine depicted in **Table 1** proved to be suitable [20].

Holes were drilled into the specimen (see **Figure 1a** and **2**) for securing in the straining holder. The following procedure had proved its value: The prepared flat specimen was located and fixed between a steel and an acryl-glass block. 1.35 mm diameter holes were drilled in the specimen through a specified opening in the fixture. These holes later served to anchor the specimen in the straining holder.

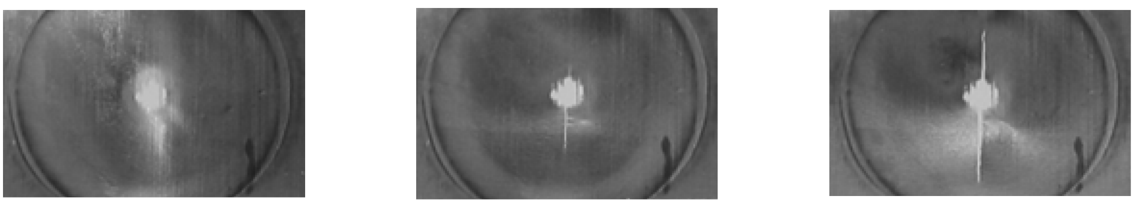
Table 1. Preparation methodology for manufacturing electron-transparent TEM tensile specimens

No.	Process step	Preparation details
1	Specimen cutting	• Wire erosion of 400 µm thick strips • Cutting to 11 mm x 2.5 mm
2	Grinding	• SiC paper • Abrasive grades from 500 to 4000 • Thickness after grinding approx. 30 µm
3	Polishing	• Contact pressure 15 N to 20 N • Rotating speed 150 rpm • Diamond paste 6 µm / 3 µm / 1µm with lubricant • Final OPS-polishing • Specimen thickness after polishing approx. 20 µm
4	Electrolytic polishing using TenuPol-5	• Electrolyte with 12 % perchloric acid (40%), 44 % butoxyethanol and 44 % acetic acid (100%) • Current strength 30 mA/cm² to 100 mA/cm²
5	Ionic polishing using Duo mill Gatan 600	• Angle 5° to 10° • Polishing duration: 10 min to 15 min



a b

Figure 1. The Gatan 654 straining holder used for the in situ TEM tests: *a* – general view; *b* – holder with the specimen



a b c

Figure 2. Macroscopically discernible crack growth during tensile loading: *a* – non-loaded specimen; *b* – crack growing with increasing load; *c* – grown crack just prior to specimen failure

Analysis of the microstructural changes during a tensile test in the TEM

A tensile specimen of the DC 04 deep-drawing steel, which was prepared using the method described above, was stepwise tested to fracture using the straining holder *Gatan 654* (see **Figure 1b)** in the TEM column. The deformation occurred at room temperature and with a constant displacement rate of 5 nm/s. **Figure 3** depicts TEM images of the transparent specimen edge, which is orientated at 90° to the tensile direction, for stepwise increasing tensile loads. It can be determined from the images that material is plastically deformed. Indications of this are a multiplication of the individual dislocations or their movement and the changes in position of the extinction contours. The horizontal separations between the microstructural elements, measured using the images, enable the local plastic strains in the observed region to be estimated. The mean value is approx. 26 %.

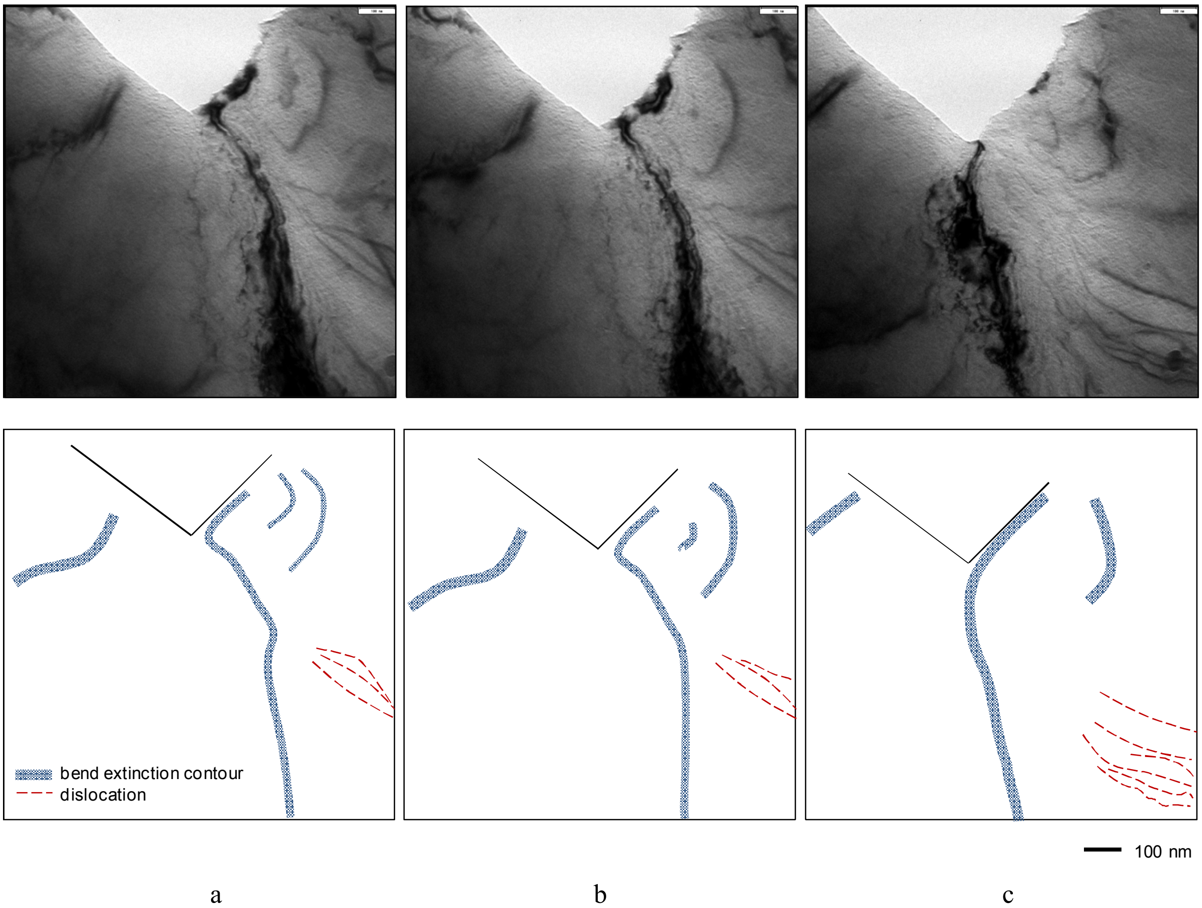


Figure 3. TEM images located at the transparent edge of a hole and schematic representation of the widening of bend extinction contours and the dislocation movement as a consequence of the plastic deformation. Increasing strains from *a* to *c*

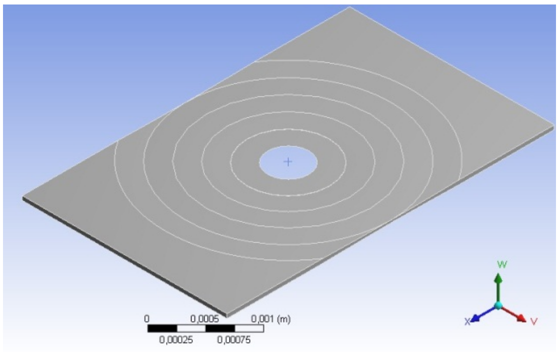
Numerical modelling

Owing to the specimen's straining, stresses arise in the material during the tensile test and the specimen deforms. The microstructural changes resulting from the straining and the crack formation can be observed at the dislocation level by using the TEM.

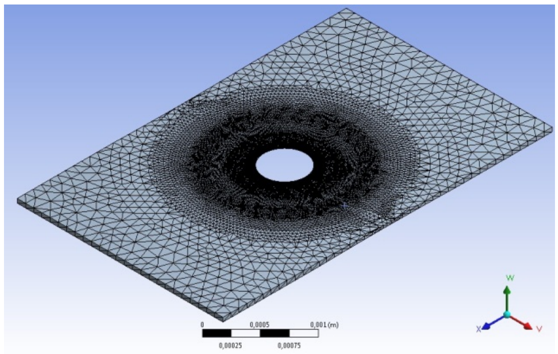
Knowledge of the stress magnitudes at which the material begins to deform is necessary to completely understand and qualitatively describe the observed effects. In addition to this, the associated deformation must be known. These can be computed by using the mathematical modelling based on finite element methods. Moreover, by modelling the tensile test of an actual specimen enables the location of crack initiation to be determined. Knowledge of this location permits one to specifically observe material failure directly at the source of failure. A comparison of the computed results, including the beginning of plastic deformation and the location of crack initiation, enables the scaleability of the macroscopically known material parameters to be verified for this type of thin material specimen. The scaleability is related to the emission or absorption of dislocations since the reduced specimen thickness is merely 50 nm to 200 nm and therefore an increase in the yield stress must be expected. Suitable software for modelling such a tensile test is the program ANSYS 13.0. The program enables the plastic deformation in the specimen, which result from the external forces, to be computed.

To assess the applicability of this methodology, a specimen geometry was selected that resembles the size and geometry of experimental specimens which are used for TEM tensile tests (see **Figure 4**). To reduce the computation time, only the central region of the specimen, containing the ion-etched hole and the flat surface, was modelled.

The surface, which was reduced by means of the ion-beam, was modelled using conically sectioned annular elements whose diameters reduce towards the centre (see **Figure 4**). In this way, the annular elements simulate the irregular material removal which occurred during the ion-etching. The finite element mesh consists of tetrahedral elements. The element size increases with increasing flattening of the specimen (see **Figure 4b**).



a



b

Figure 4. Specimen dimensions (3.5 mm × 2.5 mm × 50 µm); reduced region (10 nm to 200 nm): *a* – geometric model; *b* – FE mesh

An elastic-plastic deformation model incorporating a poly-linear, kinematic strain-hardening was employed for the computation. This can be used since the deformations in the specimen are comparatively small. In this case, a model possessing kinematic strain-hardening very accurately describes the observed process. The FE-software ANSYS employed here uses the model of Besseling taking into account the Bauschinger effect. The material parameters implemented here are based on experimental data resulting from tensile tests performed on the DC 04 deep-drawing steel considered here. The tensile movement was modelled by applying opposing displacements as boundary conditions at the two opposite specimen edges.

Simulation results

The numerical computations, based on the developed model, permit the stress and strain states to be qualitatively identified in all regions of the TEM specimen. Thus, for example, the evolution of the von *Mises* stresses is represented in **Figure 5** during the increasing specimen displacement. The simulation results show that plastic deformation occurs following a cross-head displacement of 1 µm and are localised near to the hole at 90° to the tensile direction at the outset of the test. As the deformation advances, the plastic zones spread towards the specimen's free edges. The result agrees with the observation of microstructural evolution in the TEM described above. Owing to the test piece’s geometry, the deformation state is three-dimensional. The specimen symmetrically necks to the hole. Two rigid zones exist above and below the hole. These zones remain barely deformed up to the material's failure. In practical terms, this information is helpful for introducing tags into specific low-deformation regions of the specimen at a later stage. Using these tags will enable the total cross-head displacement to be directly and precisely measured. After extending the specimen by 60 µm, the values of the ultimate tensile strength and/or the fracture strain are reached immediately adjacent to the hole's edge. This corresponds to the conditions for which crack formation and crack growth is initiated towards the TEM specimen's free edge (cf. **Figure 1**).

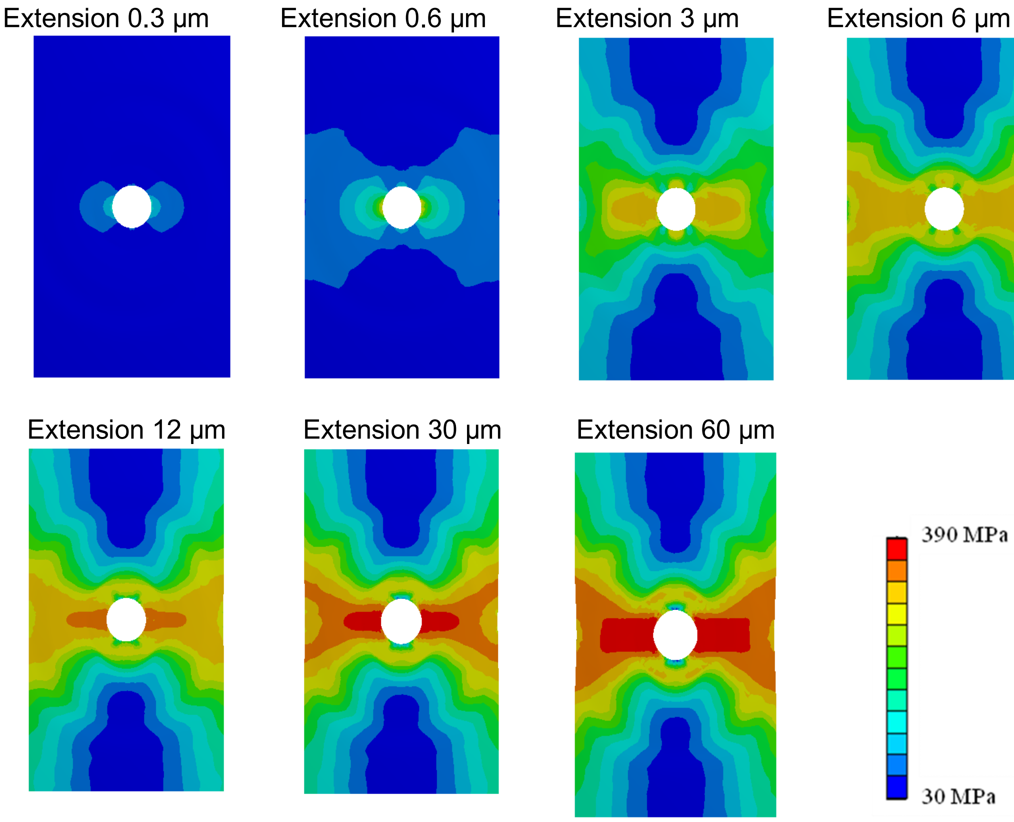


Figure 5. Distribution of the von Mises stresses resulting from increasing tensile deformation

The change in the equivalent strains from the hole's edge to the free edge midway along the specimen was analysed at an extension of 50 µm by using the numerical computational results (see **Figure 6**). It can be determined from the curve's profile that, corresponding to the stress field, the maximum strain is concentrated near to the hole. The equivalent strain is almost constant at approx. 0.22 up to a distance of 0.4 mm from the hole's edge. This value then rapidly decreases for larger distances. The calculated equivalent strain deviates by 15% from the measured strain value, which is roughly estimated from **Figure 1**. The plastic deformation component dominates the elastic deformation which is observed in the specimen's section.

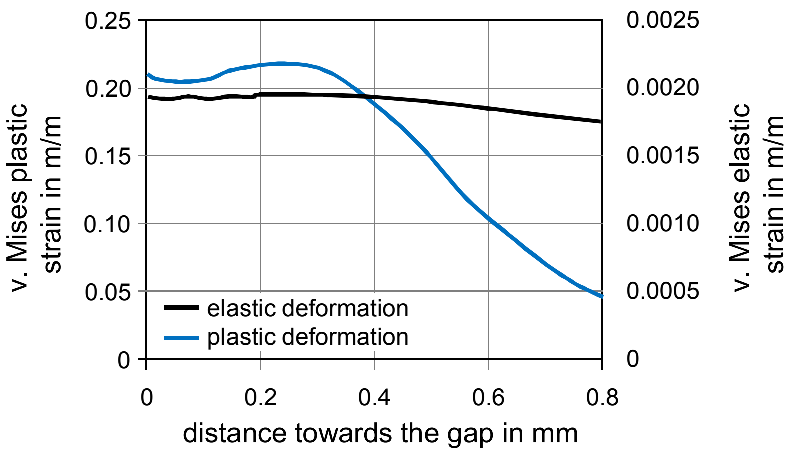


Figure 6. Change in the equivalent strains midway along the TEM tensile specimen transverse to the tensile direction

The effects observed in the simulation show that the developed model produces valuable results and can be used for investigating the specimen's behaviour during tensile tests in the TEM. The model enables the stress values for different extensions to be determined at every point in the specimen. In this way, the link between the computed data and the microstructural changes observed in the TEM lays the basis for an enhanced analysis of damage behaviour by means of in situ investigations.

Summary

The effect of the material's microstructure resulting from elastic-plastic strain can be investigated at the dislocation level by means of tensile tests in the transmission electron microscope. Here, the complicated specimen geometry, which depends on its preparation, produces an inhomogeneous distribution of stresses and strains which can not be analytically computed. Moreover, the stress and deformation fields change during the in situ tensile test. An elasto-plastic model was developed and a finite element software was implemented to determine a correlation between the displacement of the tensile straining holder and the locally operating stresses and strains in the observed material region. The results of the numerical computations show that the deformation is localised adjacent to the thinned hole region of the TEM tensile specimen. The plastic component of the deformation dominates the elastic component up to a distance of approx. 0.4 mm from the hole's edge. A comparison of the computed results with the experimental data from the in situ tensile tests in the TEM validates the developed model.

Individual TEM specimen geometries are to be considered in the future for the numerical computations. For this purpose, the actual geometry of the tensile specimen's thinned region is to be firstly optically measured using a laser microscope and then subsequently transferred into the FE-software ANSYS as a model.

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Численное моделирование испытаний на растяжение, проводимых в трансмиссионном электронном микроскопе

Нюрнбергер Ф., Столбченко М., Гридин А.,
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Изменения микроструктуры и разрушение материала вследствие упруго-пластической деформации могут быть исследованы при испытаниях на растяжение в трансмиссионном электронном микроскопе. Вследствие нестандартной формы образцов, обусловленной методикой их изготовления, невозможно точно определить значения напряжений и деформаций в наблюдаемой области. В связи с этим была разработана математическая модель для получения при помощи численных методов соотношения между удлинением образца и напряжениями и деформациями, возникающими в области наблюдения. При помощи ПО на базе метода конечных элементов проведено моделирование изменения формы образца при его растяжении, в результате чего получены значения напряжений и деформаций, соответствующих определенным шагам удлинения. Таким образом можно сопоставить изменения микроструктуры, наблюдаемым в образце в ходе испытания на растяжение в трансмиссионном электронном микроскопе, с величиной локальных деформаций и напряжений.