Probing the surface and bulk mechanical properties of NiTi shape memory alloys

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A few words about Chemnitz, its university and our research at IWW
Chemnitz University of Technology

- >10.000 Students
- 8 Departments (Mech. Eng. 26 Full Professors)
- > 50 BSc/ MSc courses
- 1 Collaborative Research Center (IWW)
- international students from more than 50 countries
- Smart Systems Campus: Fraunhofer-Institutes IWU, ENAS and IZM
- „Saxon Excellence Research“ eniPROD; TUC + FhG
- Federal Cluster of Excellence MERGE
Department of Materials Science and Engineering (IWW)
Composite Materials

Electroplating

Modelling

Ultrafine-grained Materials

Brazing

Microstructural Analysis

Thermal Spraying

(Dynamic) Materials Testing

Corrosion and Tribology

Modelling

Ultrafine-grained Materials

Corrosion and Tribology

(Modeling)

(Microstructure analysis)

(Dynamic) Materials Testing

Corrosion and Tribology

Thermal Spraying

Brazing

Electroplating

Composite Materials
Research collaborations with industry

Our alumni work, among others, with
Audi, Mathys Orthopädie GmbH
Thyssen, MTU, Enthone-OMI
Thielert Aircraft Engines, Modine
Daimler, Hilti, Airbus, AMD
Volkswagen, Salzgitter Stahl AG

Degree Courses
• Mechanical Engineering/Production Technology
• Automobile Production, Medical Engineering
• Focus Areas:
  Materials Engineering, Surface Technology, Lightweight Construction

Further Activities
• Seminars
• Further training courses
• Post-graduate programmes
• Student projects
Research areas (general area of mechanical properties and microstructure)

- NiTi shape memory alloys, martensites
- Al, Mg, Ti
- High strain rate deformation, shear bands
- Localization of deformation
- Thermo-mechanical optimization
- SPD: ECAP, cryo-forming

Methods, tools

- Mechanical testing, including strain mapping, thermal imaging
- Microstructural analysis (SEM, TEM, XRD...)
- High strain rate testing (drop tower, rotating wheel, Split Hopkinson)
- ECAP (different tools, 16 MN press)
- Modeling: atomistic, micromechanics, FEM
DFG PAK250: Biaxial compression testing
Herstellung der Faserbeschichtung

Herstellung der Schicht-Fasersysteme bzw. des Verbundes

Charakterisierung und Modellierung

Mechanische Charakterisierung (statische, zyklische, dynamische Beanspruchung, Kriechen, Bruchmechanik)

Charakterisierung der Systemeigenschaften (Verschleiß, Alterung / Korrosion, Haftung)

Modellierung und numerische Simulation

Transfer (ökonomisch/technologisch)
Ecological and economical machining

MICROPLAST

DFG-FOR797

DFG-PAK292

Prinzip Bohrungsdrücken
Collaborative Research Center SFB 692

Optimization of high-strength aluminum alloys:

a) ECAP deformation and thermo-mechanical processing

b) Aluminum matrix composites

c) Al-Mg-composites
The team of SFB 692, September 2013
• „Materials that you can deform and that, when heated, recover their initial shape“

• Potential use (to be evaluated critically): healing dents in a car body, morphing wings, alternatives to complex motors and gears, sensor and actuator capabilities in one single part

• Note: „Any sufficiently advanced technology is indistinguishable from magic....“ (A.C. Clarke)
ISI Web of Science

„NiTi OR Nitinol“ → 3513 search results, 1945 – 2007
„SMA“ → 7464 search results

masses of papers

papers „with class“
„If you want to teach materials science – teach shape memory alloys“

General class of materials: ferroics, domains

- CuZnAl:
  - $\alpha^+ - \alpha^-$ martensite

- FeNiAl:
  - “fractal“ martensite

- $\alpha$-Fe:
  - Ferromagnetic domains

- BaTiO$_3$:
  - Ferroelectric domains

E. Hornbogen, Int. J. Mat. Res. 96 (2005), 316-324.
Rearrangement of domains...

(a) B2 → B19'

(b)

(c)

(d)
Analogies

ferro-

elastic
martensitic
~ 10 %
temperature, stress
large forces, but slow

magnetic
ferromagnetic
~ 1 %
magnetic field H, stress
small forces, fast

electric
ferroelectric
~ 0.1 %
electric field E, stress
large forces, fast

materials

(stimuli) shape memory alloys: Cu-based, Fe-based, NiTi
Why do research on NiTi?

„The only successful shape memory alloy is the one least understood.“

X. Ren, Bochum, 2003
SM Effects – Overview: Thermal SM

Engineering science, applications: (no paper clips!) actuators, thermostats Forces, actuation frequency, design standards

“one-way effect“
Effects II: Pseudoelasticity (mechanical memory)

Focus on biomedical applications

Clausius-Clapeyron type relationship between (ambient) temperature and transformation stresses

\[ c_{AM} = 5 - 15 \text{ MPa} / \text{K} \]
Effects II $\frac{1}{2}$ : two-way effect

Requires „training“, fatigue, low (almost zero) forces allowed
SMA applications

1. Fashion, decoration, gadgets
2. Couplings and fasteners
3. (Micro-) Actuators
4. Adaptive materials and hybrid composites
5. Damping apps
...


Medical Applications (see paper by Duerig et al.)
1. Stents
2. Guide wires
3. Vena cava filters
4. Orthodontic drills and braces

Medical market dominated by NiTi
Stents: fine structures and cyclic loading

FDA: $4 \times 10^8$ cycles
A non-linear drilling device
Examples of applications: Thermostat

(c) Eberspächer
Examples of applications: Thermostat

10 minutes
15 minutes
20 minutes

(c) Eberspächer
Limitations and Issues
Higher symmetry of austenite: all martensite variants have to return to the one distinct austenite configuration.
Martensitic Transformations

historically: Fe-C, tetragonal distortion
today: ALL diffusionless phase transformations
- chemical composition remains unchanged
- cooperative shear deformation in the lattice, lattice constants change only a little
- nearest neighbors remain the same
- considerable deformation, up to 15 % in single crystals
- habit plane between austenite and martensite
- well-defined orientation-relationships
Important NiTi crystal structures

Martensitic transformation in SMAs

(a) B2' B19'
(b) TiAl, mechanical twin
(c) Self-accommodation
(d) Skrotzki, Acta Mat, 2000, 851-862
Martensitic transformation in SMAs
SM effects - again: EWE

Materials science: twinning, detwinning, nucleation, transformation...
Pseudoelasticity
Training generates lattice defects
Microstructure: recent advances

D. Norfleet et al., Acta Mater. 57(2009), 3549


TEM in-situ straining
Nanoindentation of pseudoelastic NiTi

\[ RDR = \frac{h_r}{h_{\text{max}}} \]

Janine Pfetzing
Nanoindentation and irreversible deformation

(small) Berkovich indentation

spherical indentation:
- ▲ $R = 50 \, \mu m$
- ○ $R = 25 \, \mu m$
- □ $R = 5 \, \mu m$

remnant depth ratio $\rho$ (%) vs.
$0.2 \, a/R$ (-)
Nanoindentation and irreversible deformation

Small maximum loads (RDR ~10%)

Moderately large maximum loads (RDR ~30%)

\[ RDR = \frac{h_r}{h_{\text{max}}} \]
Nanoindentation and strain gradients

Spherical: $R = 10$ microns

Berkovich

Plastic deformation occurs (almost) from the very beginning!
Even under optimized conditions: Strain gradients result in formation of GNDs.

\[ RDR = \frac{h_r}{h_{max}} \]

\[ \varepsilon^{sph} = \frac{a}{5R} \]

\[ \rho_{GND}^{sph} = \frac{r}{bR} \]

\[ \varepsilon^{Berk} = 0.2 \cot \alpha \approx 7.2\% \]

\[ \rho_{GND}^{SS} = \frac{3h_c}{2ba^2} = \frac{3}{2bh_c} \tan^2 \theta \]
2nd regime „beyond the plateau“?
Orientation effects in NiTi / NiAl
Continuous Stiffness Measurements

elastic moduli
Continuous Stiffness Measurements

- hardness

[Graph showing hardness vs. displacement for NiTi and NiAl with different crystallographic orientations.]
Micro-pillar Compression
Pseudoelastic deformation

Micro-pillar compression-Pseudoelastic strain

Buckling characterization

\[ \varepsilon_{\text{est}} = 7\% \]

Stress-strain

(i)

(ii)

Engineering stress, \( \sigma \) (GPa)

Engineering strain, \( \varepsilon \) (%)
Permanent deformation

Micro-pillar compression-video

In-situ Compression

Post deformation SEM
Summary

- Research and teaching in Chemnitz

- Introduction to shape memory alloys

- Recent results on surface testing of NiTi:
  - Nanoindentation is related to irreversible deformation
  - Indenter tip geometry and testing conditions affect reversibility
  - Orientation effects are due to the martensitic transformation
  - Micro-pillar studies need in-situ observation.