Thermo-mechanical finite element modeling of shape memory materials’ microindentation

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Abstract. Indentation of shape memory materials and later heating with recovery of indent is studied in this work using finite element modelling. Results of simulations of two types of shape memory materials, with one-way shape memory effect and with superelastic properties compared to experimental indentation with 200µm spherical indenter. Based on results of finite element modeling, several useful quantities plotted for loading, unloading and thermal recovery for various materials with shape memory effect. Recovery of imprint made with Berkovich (three-sided pyramid) compared to recovery of imprint made with spherical indenter.

1. Introduction
Investigation of martensite in 1900s and later the thermally reversible martensitic structure in CuZn and CuAl alloys [1] was a big step for future discovery of “smart” materials and the most significant among them, shape memory materials (SMM). Compared to standard materials they have many extraordinary properties. The most interesting is what they can, as it can be guessed from the name, remember their shape before deformation occurred. The specimen, deformed unrecoverably can restore original form after heating. Also, at certain temperatures they can acquire superelastic properties and deform recoverably to bigger degrees compared to stainless steel. This potential utilized in many fields including aerospace, biomechanical, in nanotechnology as sensors and actuators.

Shape memory alloys (SMAs) can exist in two different phases (austenite and martensite), martensite phase can also have two different structures (twinned martensite, detwinned martensite). This arrangement is possible at different temperatures and stress states depending on principle of minimum free energy and forms six different possible transformations [2]. Such transformations not carried by diffusion but rather by shift of crystal lattice [3]. In summary, we can classify SMA behaviour in three categories.

1. Superelasticity (SE) or Pseudoelasticity (PE) – recoverable deformation up to 10% [4] without change of ambient temperature. This exceptional deformations achieved not by elastic change of interatomic spacing but due to phase transition. Usually a significant hysteresis presents. Stresses of forward transformation start and finish can be much higher than of reverse transformation.

2. One-way shape memory effect – deformation of material can be recovered only after heating. After withdraw of applied force deformation still presents and material can be restored to original shape by heating. Following cooling does not result in change of geometry or volume.

3. Two-way shape memory effect – heating and cooling results in recovery of shape of high and low temperatures. Such materials can be obtained by cyclic deformation training. One cycle includes
deformation at required temperature, unloading, heating and cooling until demanded shape is achieved [5]. As reported, usually only half of one-way shape memory effect strain can be attained [6].

Many applications of SMAs are of very small scale. Such as surgical instruments and stents [7], micro-actuators [8], medical implants [9]. Such small parts require advanced methods for testing of mechanical properties. One of the most widely used techniques for determination of mechanical properties in micro- and nano- scales is instrumented microindentation. During this test, an indenter (usually a pyramid or a sphere) is pressed to material with increasing load. Material response of displacement versus load (P-h diagram) is recorded and analysed. For classical materials without phase transformation a Young modulus can be obtained with high degree of confidence [10]. Several methods exists for determination of yield stress and hardening exponent [11]. Shape memory materials have more complicated behavior including phase transformation, temperature dependence and Young moduli for different phases. As result, the problem of obtaining mechanical properties from indentation results is solved in full. Indentation is a complex loading and it can be very hard to divide one phase from other and from phase transformation process. This paper analyses process of indentation of SMAs, thermal recovery using finite element method and discusses possibilities for retrieving mechanical properties with instrumented indentation.

2. Methods and materials

Finite element modeling (FEM) was utilized to study indentation of shape memory materials with 200 micrometer spherical indenter. Since the testing is axisymmetric, the two-dimensional FE model can be used without loss of accuracy with fitting boundary conditions. Modelled indenter, made from zirconium dioxide and SMA material up to 1000 micrometer from indenter’s tip in surface and in depth. Contact’s formulation between material and indenter includes friction effect, friction coefficient is taken 0.2. There are 2526 eight-node elements representing material and indenter in model (Figure 1). Nodes in point of initial contact are merged for better convergence and to avoid slipping of indenter to material. As usually, in contact area and in volume of bigger gradients of stresses and deformations the mesh density is higher for more efficient simulation analysis.

![Figure 1. FE model of material (blue) and indenter (grey) produced in specialized software. Image to the right enlarged for illustrational purposes.](image)

SMA has a rather unique behaviour. They can deform elastic to some degree, after that with increasing load the phase transition occur and new phase can deform elastic also. When load it removed, material can ether return to original shape or recover only elastic deformation, depending on temperature. At some conditions, only the change of temperature results in phase transition and change of shape. Evidently, such materials require own model with much higher degree of complexity compared to most elastic and plastic models. The constitutive model for shape memory effect simulations used for this work proposed by Auricchio et al [12-13]. This is a phenomenological constitutive model
based on continuum thermodynamics with internal variables. It can represent such thermomechanical behavior as shape memory effect, superelasticity, different response to tension and compression.

\[
\Psi(\varepsilon, T, \varepsilon_{tr}) = \frac{1}{2} (\varepsilon - \varepsilon_{tr}) : D : (\varepsilon - \varepsilon_{tr}) + \tau_M(T) \| \varepsilon'_{tr} \|^2 + \frac{1}{2} h \| \varepsilon'_{tr} \|^2 + I_{\varepsilon'_{tr}}(\varepsilon'_{tr})
\]  

(1)

where \( D \) - material elastic stiffness tensor, \( \varepsilon \) - total strain, \( \varepsilon_{tr} \) - total transformation strain, \( \varepsilon'_{tr} \) - deviatoric transformation strain, \( \tau_M(T) \) - a positive and monotonically increasing function of the temperature as \( \langle \beta(T - T_0) \rangle^+ \) in which \( \langle \cdot \rangle^+ \) is the positive part of the argument (also known as Maxwell stress), \( \beta \) - material parameter, \( T \) - temperature, \( T_0 \) - temperature below which no austenite is observed in a stress-free state, \( h \) - material parameter related to the hardening of the material during the phase transformation, \( \| \ldots \| \) - indicator function introduced to satisfy the constraint on the transformation norm.

Two NiTi alloys with different compounds were studied for this work. One of them, 55.9 Ni 44.1 Ti is superelastic at room temperature, and the other, 55.1 Ni 44.9 Ti can deform at room temperature and recover original shape when heated above 75 °C. Indentation testing performed at room temperature, while simulation temperatures varied. Physical properties presented in table below (Table 1).

**Table 1.** Transformation temperatures in degrees Celsius and stress at which transformation starts (for alloy at \( M_s \) temperature) [14].

| Alloy            | \( M_s \) | \( M_f \) | \( A_s \) | \( A_f \) | \( \sigma_0 \) |
|------------------|----------|----------|----------|----------|-------------|
| 55.9 Ni 44.1 Ti  | -40      | -57      | -25      | -5       | 85          |
| 55.1 Ni 44.9 Ti  | 18       | -22      | 35       | 73       | 100         |

**3. Experiments and simulations**

Finite element modelling method allows to change material constants and thus the material behaviour depending on constitutive model. In this particular case for each material it lists elastic moduli for austenite and martensite phases, stress at transformation start at temperature \( M_s \), transformation hardening constant, maximum transformation strain, hysteresis and several thermal constants. Simulation of materials listed in table 1 compared to experiments of indentation to check the consistency of FE model used. Results on picture below (Figure 2).

**Figure 2.** Comparison of experiments and simulations performed. Experimental data taken from averaging of series of indents.

Error can come possibly from inaccuracy in determination of mechanical properties, unevenness of prepared surface, differences of model behaviour and real material deformation and reformations resulted in change of mechanical properties of materials due to discharge of heat in martensite transformation.
Numerical results of simulation can be used to plot areas of material state during loading. At maximum load (Figure 3) this result is similar for both types of materials. Full transformation is observed at 1/10 of indenter’s radius below indenter’s apex until 1/4 of radius with maximum stress in the middle of this area. Also, we can mark transient area where both phases are present and detwinning/phase transformation process is not fully completed. This material state corresponds with stress plateau during uniaxial extension tests.

![Figure 3. Division of material to different phases during indentation at maximum load (5N). This is axisymmetric cut of model and left line of material shown is axis of force application to indenter.](image)

4. Recovery of microindents
When indent is made below A_f temperature it may not fully recover after the load is removed. Part of material under print still in martensitic phase and thermodynamic condition are so it requires energy to return original shape. With increasing temperature the energy of crystal lattice is also increasing and reverse transformation become possible. Studies show what smaller spherical indents compared to radius of indenter sphere, usually recovers [15]. On other hand, indents made with pyramid indenter (Vickers or Berkovich) do not fully recover due to rotation of structure (Figure 4).

![Figure 4. Pictures of one indent taken before heating the specimen (left) and after (right). Indented material - 55.1 Ni 44.9 Ti at room temperature.](image)

Process shown on the pictures above can be modelled with finite element method. This requires coupling of two different solvers with different element formulation and different degrees of freedom. For deformable body we have forces and displacements while in thermal problem it will be temperature. Thermal problem is rather simple: uniform change of temperature is supposed. Because both problems share same model and topology, temperature values over time for every element can be transferred to deformable body solver as a thermal load. Change of temperature lead to change of material behaviour according to constitutive model used and eventually, the recovery of imprint.

Initial loading take place at -10°C temperature at which material shows one-way shape memory effect. As expected at this temperature, material can recover only partially. Later, the temperature is in-
creased and material recovers. Third axis can be added to standard $P$-$h$ diagram to show recovery upon heating (Figure 5). This agrees with obtained experimental data for indentation with spherical indenter and with other works [15]. Coefficient of thermal expansion is set to $8 \times 10^{-6}$ °C$^{-1}$ so effect of volume increasing with temperature affect the results less than 1% and can be neglected.

![Figure 5](image)

**Figure 5.** Simulated $P$-$h$ diagram with temperature component. Indentation made with spherical 200 µm indenter. Increasing of temperature follows full unloading.

Furthermore, it is possible research indentation process and thermal recovery not only on changes in $P$-$h$ diagram which can be obtained using modern NI machines but also on changes of volume of different phases during loading, unloading and following change of temperature. This can be of greater interest because such graph cannot be obtained using current technologies.

![Figure 6](image)

**Figure 6.** Scaled to maximum volume of each phase over time in indentation process. Volume of initial phase is virtually infinite as it depends on volume of specimen. First 100 secs is loading and unloading and following 100 secs is thermal recovery of imprint.

For simplicity, two dimension plot is used for dependence of each phase volume vs. time (Figure 6). There are also two graphs of temperature and force applied to indenter with respect to time.
5. Conclusion
Indentation of shape memory materials and their thermal recovery is researched using finite element modeling with constitutive thermodynamical model. Results of simulations for OWSME and SE compared to indentation of NiTi alloys with different composition so they have such effects. Using results of simulation, volumetric composition and areas of different phases and structures.

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