Vacuum brazing and heat treatment of NiTi shape memory alloys

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Abstract. The pseudoelasticity of NiTi shape memory alloys is a unique material property which can be characterized by a complete recovery of a previously impressed component shape by a change of the thermal or mechanical load conditions after deforming. In contrast to the elastic deformation of ordinary materials like steels, twentyfold higher elastic strain rates up to 10 % are possible due to a temperature or a stress induced diffusion-free transformation of the crystal lattice between the austenite and martensite phases. Therefore, these superelastic alloys are frequently used as actuators, implants or stents so that there is an extraordinary high requirement of reliability and biocompatibility. In terms of joining, vacuum brazing might be a particularly suitable method to produce joined components which preserve a maximum of pseudoelasticity. Within the present research, it was shown that the vacuum brazing process at 1180 °C using pure niobium is well integrable into a solution annealing and a shape annealing heat treatment in a single furnace run. This led to a distinct tension plateau at around 285 MPa with an almost R-phase-free conversion of NiTi. Furthermore, it was proven that the share of the superelastic and proeutectic NiTiNb-phase was significantly increased with the dwell time.

1. Introduction
Shape memory alloys are metals that are able to “remember” their original shape. The mechanism of the memory effects is a change of the crystalline structure depending on temperature or stress level. The high temperature phase is austenite while the low temperature phase is martensite. The latter can occur in a twinned or detwinned structure. There are three shape memory effects: one-way memory effect, two-way memory effect and pseudoelasticity.

One-way effect: The crystal structure at room temperature is the twinned martensite. When mechanical stress is applied the martensite gets detwinned so that a plastic deformation of the component occurs. A subsequent heat treatment (above austenite-finish temperature) leads to a change of the crystalline structure to the high temperature phase austenite. This is accompanied by a deformation to the original shape of the component. During the subsequent cooling, the twinned martensite is formed without further deformation.

Two-way effect: In this case a change of temperature leads to a deformation of the component. Each of the crystalline structures (martensite and austenite) has a different shape. To obtain this effect, the component has to “learn” the two different shapes using a shape-set heat treatment.

Pseudoelasticity: The crystal structure at ambient temperature is the high temperature phase austenite. During the application of mechanical stress, the austenite transforms into martensite. After a stress relief the martensite transforms into austenite and the original shape is obtained. This behavior
leads to a tension plateau in the stress strain curve which results in high reversible strains up to approx. 10 % [1-4].

Beside the austenitic and martensitic phase, a so-called R-phase can occur. This phase is martensitic and forms due to precipitations or cold deformation. The presence of the R-phase increases the transition temperature due to a previous transformation to austenite [5-7]. The most common shape memory alloys are NiTi materials which are also biocompatible. To obtain the shape memory effects the ratio of Ni and Ti is essential. Small deviations of the chemical composition can change the material properties such as transformation temperatures and the shape memory effect. The applications of the shape memory alloys are especially the medical and actuator technology [3, 4, 8-10].

Joining of shape memory alloys is still today extremely challenging since the main requirements for the joint is to preserve the maximum of the shape memory effects as well as to assure a high biocompatibility [3, 4, 8-10]. In addition, there is a highly limited range of suitable parameter with regard to the alloy composition and in particular to the heat treatment. Adhesive bonding, clamping and crimping are used if there is just a low demand to the strength and the joint is not part of the actual functionality [8, 9, 11, 12]. The following statements refer to NiTi shape memory alloys, but are generally also valid for other ones. Today, laser welding is state of the art and frequently used to obtain high strength joints of shape memory alloys [13-26]. This is mainly due to a very high energy density which is furthermore extremely accurate to the local position so that the total heat input as well as the size of the heat affected zone (HAZ) are well controllable. In the HAZ, there is just a slight grain growth while the weld metal has a coarse-grained and cast-like structure [13, 17, 18]. As a consequence of this, other welding technologies like WIG or plasma welding are only used sporadically because the size of the HAZ and the weld pool are usually larger which affects the shape memory affects significantly [15, 25, 26]. However, laser welding of shape memory alloys is extensively researched but the joint geometry is technically limited to wire-to-wire or to butt joints. Furthermore, the size of a laser welded joint is between 0.5-4.0 mm and as a consequence of this, the joints are not able to obtain the maximum possible expansion rate of the base material [14, 16, 20]. In detail, this results from the precipitation of some martensitic phases in the weld metal during solidification within the regular austenitic microstructure. During loading these martensitic phases are detwinned and thus, dislocations will be formed which avoid a complete martensite to austenite retransformation on load reduction below the tension plateau. In addition to this, brittle phases such as Ti: Ni and Ni: Ti are frequently present in the weld metal [14]. Both aspects lead to an irreversible elongation even if there is just a load applied to the component which is normally in the elastic range for shape memory alloys [20]. Nevertheless, laser welding is well suited to produce high strength joints of NiTi shape memory alloys when considering these explanations for the design of the component.

From another point of view, vacuum brazing might be able to preserve a significant higher level of pseudoelasticity than laser welding since the thickness of the joint is just below 0.1 mm in general. In brazing it is well known, that the strength of a joint is highly enhanced if the thickness of the braze metal is near the zero gap [27, 28]. Due to this, it is imaginable that this effect on the strength is possibly transferable to the pseudoelasticity of shape memory alloys and in particular if the braze metal itself consists of superelastic phases. Vacuum brazing offers the advantage to connect large areas or even several joints at the same time and unite the joining and the heat treatment in a single furnace run.

However, before discussing the vacuum brazing in detail, the heat treatment on the shape memory alloys will be described extensively hereafter. Due to the high oxygen affinity of titanium, NiTi shape memory alloys are usually produced by induction or arc melting in vacuum systems. After this, an optional homogenization annealing at approximately 1000 °C can take place before hot forming (open-die forging, hot rolling) between 600-900 °C and subsequent cold forming (cold rolling, wire drawing) which is carried out to generate a specified amount of dislocations. In this condition, the material features poor mechanical properties and almost no shape memory effects [29, 30]. A fine-grain microstructure is achieved by multiple cold forming with particular specific degrees of deformation and intermediate annealing. A final shape-set heat treatment under load between 350-550 °C enables the shape memory properties to be set in a defined manner. In detail, the remaining dislocations and Ni:Ti: phases were
reduced specifically by the occurring recovery process. Thus, the remaining amount of Ni₄Ti₃ precipitations is a function of the heat-treatment and as a consequence of this the austenite-finish temperature and the strength level of the tension plateau are adjustable for each individual shape memory alloy [29, 31]. The Ni₄Ti₃-phase is thermodynamically stable between 300-500 °C so that it is practicable to use a shape-set temperature of 510-530 °C [29]. As explained before, this martensitic R-phase will primarily transfer to austenite on very small load application so that there is at first a non-linear response. Anyway, this finally results in a very small pseudoplastic elongation what can easily be considered in technical applications. It should be noted that a heat treatment above the maximum shape-set temperature of 550 °C is barely investigated with respect to pseudoelasticity and only applied by very few scientists in terms of vacuum brazing [32-36]. Due to the necessity of a total base material heat treatment during vacuum brazing, a solution annealing process between 830-1180 °C of the base material is automatically carried out with the brazing. As a result, previously precipitated Ni₄Ti₃-phases which impair pseudoelasticity are brought into solution in the nickel mixed crystal and suppressed in precipitation by rapid pressure gas quenching during cooling [37-39]. Compared to laser welding, a vacuum furnace process is a fairly slow process so that there will be a noticeable grain growth of the originally ultra-fine-grained shape memory alloy. This can also cause an irreversible expansion of the components [30]. It is vital to know for the present research work, that the grain growth is primarily increasing with temperature and time but is slowed down by the total amount of Ni₄Ti₃-phases as well as by dislocations [1, 30].

Vacuum brazing of shape memory alloys as well as the effect of solution annealing on the pseudoelastic behavior is still today not investigated adequately by far. There is some literature for which pure copper was used in terms of infrared brazing NiTi [36]. In detail, the strategy was to substitute the nickel of the shape memory alloy by copper due to the formation of Ti₅₀Ni₅₀-xCuₓ (x ≤ 30 wt.-%). It was shown that the presence of the Ti(Cu,Ni)-phase, which is considered superelastic, increased with the dwell time. However, the most relevant research on brazing NiTi shape memory alloys is based on using niobium as a brazing filler metal. As well as NiTi, niobium exhibits biocompatibility which is essential for the use in the medical technology. Grummon et al. and Wang et al. investigated the brazing of NiTi joints using niobium as brazing filler metal. In figure 1 the quasi-binary NiTi-Nb phase diagram is shown.

![Figure 1. Quasi-binary NiTi-Nb phase diagram [32].](image-url)

At niobium content of 26 at.-% at 1170 °C, there is a eutectic point. The vacuum brazing process of Grummon et al. were conducted at a temperature of 1180 °C with different dwell times (1 s to 6 min)
using a pure niobium foil (thickness: 50 µm). A subsequent heat treatment was conducted at 350 °C for 90 min. The microstructure of the joints consisted of proeutectic NiTi dendrites as well as a eutectic NiTi-Nb phase (figure 2). As one can see for the horizontal line at 1180 °C, there is a solid-liquid equilibrium for the phases of concentration C1 and C2. As a consequence of their different niobium content there is a bidirectional diffusion leading to the liquid quasi-eutectic phase.

Figure 2. SEM images of NiTi brazing joints with Nb as filler metal alloy. Different dwell times at a brazing temperature of 1180 °C [32].

There was a tensile strength of the joints of about 800 MPa and a tension plateau in the stress strain curve was proven after brazing. Wang et al. used a NiTi-Nb powder (ratio 3:2) for brazing with a brazing temperature of 1180 °C (4–8 min) and a subsequent heat treatment at 530 °C (30 min). Comparable to the results of Grummon et al. the microstructure consisted of a proeutectic and a eutectic phase [32-34, 40].

The main objective of the project is to evaluate the effects of a vacuum heat treatment and vacuum brazing process on the microstructure and pseudoelasticity of NiTi and the joint properties as well with special regard to the pseudoelasticity. Since there are very few and hardly any studies on these interactions during vacuum brazing of NiTi, the present research is characterized to give an evidence of feasibility. As a matter of course, the first task is to investigate the effect of a solution annealing process in a vacuum furnace on the base material properties. In foresight to limit the expected increase in grain size during this process, it might be reasonable to use a cold-formed NiTi alloy which was never done before. As a result of the initial recovery processes during heating up, an inhibition of grain growth and thus, a significantly better pseudoelasticity may be achievable. Hence, the effect of solution annealing between 800-1180 °C will be investigated for both, a regular shape-set condition as well as a cold-formed condition. In this context, a comprehensive vacuum furnace process will be designed which includes solution annealing, brazing, quenching, and shape-set. In addition, NiTi shape memory alloys will be brazed at 1180 °C for a dwell time of one second to 34 minutes by using a pure niobium foil of thickness 50 µm.

2. Experimental and materials
The heat treatment and brazing procedures were carried out in a horizontal vacuum furnace type EU80/1H (Schmetz Inc.) in a molybdenum heating chamber. NiTi-wires of diameter 1.6 mm were used for the investigations on the effect of solution annealing on the base material properties. As explained in the introduction, one set of the wires were in a regular straight annealed condition (50.5 at.-% Ni and 49.5 at.-% Ti, Imasys Inc.) whereas a second set was in a cold-formed condition with a degree of deformation of φ=40.7 % (51.5 at.-% Ni and 48.5 at.-% Ti, Ingpuls Inc.) which is in contrast to the
regular wire not pseudoelastic but ultra-fine grained. In order to prevent reactions of NiTi with the molybdenum, the wires were placed inside of an Al₂O₃ tube. After the evacuation to a high vacuum level better than 6.2*10⁻⁶ mbar, the wires very slowly heated up to 200 °C with 10 K/min and hold for 10 minutes. This was to assure an equal temperature distribution within the furnace as well as to prevent a formation of Ni₃Ti which is thermodynamically possible from 250 °C [38]. Furthermore, there is no effect of this temperature to the wires. Afterwards, a very fast heating rate of 100 K/Min was set to minimize the total heat input into the base material which was intended to limit the grain growth which is considered to decrease pseudoelasticity. Solution annealing temperatures of 800 °C and 1180 °C were investigated for a fix dwell time of five minutes. Argon was used as quenching gas with a partial pressure of 1.5 bars to avoid an extensively reaction of titanium with nitrogen what is used in general. Furthermore, the rapid cooling to room temperature was also necessary to suppress the precipitation of Ni₃Ti-phases which impair pseudoelasticity and shifts the transformation temperature. Hence, a convective cooling gas flow was used by a heat exchanger with an integrated fan. Afterwards the wires were directly shape-set annealed for five minutes at 510 °C. An exception of this was given for some regular wires which were solution annealed to 800 °C and then shape-set annealed at 400 °C for five minutes so that a formation of the R-phase is intended which will be used as a reference to show up this effect. Tensile testing was carried on an Instron Inc. testing machine type E10BMTM1020 at a test speed of 0.02 mm/s at room temperature by using an inductive displacement sensor. It is well known that the microstructural transformation from austenite to martensite is exothermic. Because the stress-strain behavior is very temperature dependent a convective ambient air flow was used to assure the comparability of the results [41]. Three wires of length 150 mm were examined for each parameter and 25 mm were used for clamping on each side.

Due to the fact that applicability of vacuum brazing is clearly intended to join larger areas, two NiTi samples of size 10x10x2 mm were used (49.5 at.-% Ni and 50.5 at.-% Ti, Euroflex Inc.). Inbetween a pure niobium foil of thickness 50 µm was placed which has a melting point of 2477 °C. As explained in the introduction, an eutectic phase in the quasi-binary NiTi-Nb system is present by 26 at-% Nb at 1170 °C. Therefore, brazing was conducted at 1180 °C by using a heating rate of 10 K/min. In order to investigate the effect of the dwell time on the microstructural composition of the braze metal, a dwell time of one second to 34 minutes was used. Vacuum cooling was set after brazing and no additional shape-set heat treatment was carried out. For tensile testing two NiTi plates of 30x20x2 mm were brazed with a dwell time of 6 min on the narrow front side. Afterwards a non-standardized flat tensile test geometry was cut out by using wire-cut EDM (Electrical Discharge Machining). The brazed samples were cross-sectioned and polished finally with a one micrometer diamond suspension. A scanning electron field emission microscope type JSM7001F (Jeol Inc.) as well as an integrated EDS-detector (Oxford Inc.) were used for the microstructural analysis of the cross-sections.

3. Results and discussion

3.1. Effect of the high temperature heat treatment on the NiTi base material properties

The results of tensile testing were shown in figure 3 for which the original state of the regular straight annealed condition is shown in a line whereas the cold-formed condition is shown in a dashed line. For reasons of clarity, only one curve is shown.

As expected, a well superelastic stress-strain behavior was proven for the common delivery condition which has a clearly tension plateau of around 500 MPa at which the austenite to martensite transformation takes place. However, there were two level of slope visible prior to the beginning of the plateau which might be a consequence of the clamping device. A maximum tensile strength of 1800 MPa was determined for the cold-formed state without pseudoelastic behavior. Through a vacuum shape-set annealing of these wires at 510 °C (curve c)), a tension plateau at around 270 MPa was achieved and thus, they had a pseudoelastic behavior now. However, there was a non-linear stress-strain relation at the beginning of load application what is characteristic for the presence of the R-phase. Based on this it can be assumed that most of the dislocations were relieved by forming Ni₃Ti. Due to the vacuum shape-set annealing, the tension plateau of the regular wires was reduced to approximately 350 MPa and a
lower maximum elastic strain was observed. Furthermore, there were still two level of slope visible at the beginning of the curve. It could be determined that a shape-set annealing temperature of merely 400 °C after solution annealing at 800 °C is not useful because the stress-strain curve indicates the presence of the R-phase (curve d). In contrast to this, a linear stress-strain behavior was found for all the solution annealed and at 510 °C shape-set annealed wires of the originally regular condition (curves e-f). In addition, it could be shown that the level of the tension plateau was increased by 35 MPa to 285 MPa, if the solution annealing temperature was increased from 800 °C to 1180 °C which means brazing temperature. A similar behavior was found for the originally cold-formed condition so that the level of the tension plateau was 305 MPa for a solution temperature of 1180 °C whereas it was just 200 MPa for the solution annealed wires at 800 °C. To summarize the results of tensile testing, it was found that the designed vacuum process is suitable to assure a pseudoelastic behavior of NiTi base materials after the brazing cycle. Furthermore, it could be proven that the approach to use cold-formed shape memory alloys for brazing might be very promising since there was a pseudoeelastic behavior afterwards. Actually, the presence of the R-phase was indicated for these samples but this can be hopefully prevented in future by increasing the shape-set temperature or the dwell time as well. The results achieved are of course not comparable to the ultra-fine grained regular condition of NiTi shape memory alloys but still show a high degree of the tension plateau and very high strain rates. The effect of the heat treatment on the grain size, on the formation of phases, and on the pseudoelasticity will be examined more detailed in future.

**Figure 3.** Effect of vacuum heat treatment on the stress-strain behavior of NiTi-wires, a) regular as-delivered condition, b) cold-formed as-delivered condition, c) shape-set annealed, d-f) solution annealed and shape-set annealed. A dwell time of five minutes was used for all given temperatures in the figure.
3.2 Microstructural analysis of niobium brazed NiTi

In figure 4 the SEM image of a cross section of a brazed joint with brazing parameters of 1180 °C and 6 min dwell time is shown. In the center of the brazing zone a NiTi-Nb eutectic is formed with a proportion of approx. 23 % niobium (see element analysis in figure 4, position 3). Thus, the brazing seam has a much larger thickness (100 – 150 µm) than the used niobium foil (50 µm). The composition of the eutectic (23 % Nb) is consistent with those in other research projects (20 – 26 % Nb) [33, 42-44]. Between the base material and the eutectic, proeutectic dendrites are present. This phase consists of approx. 7 % niobium (figure 4, position 1) which matches the solubility of niobium in NiTi at the brazing temperature of 1180 °C (figure 1).

The effect of the dwell time at a brazing temperature of 1180 °C on the microstructure is shown in figure 5. Both phases (eutectic and proeutectic) change with increasing dwell time. Due to the longer period of time at a higher temperature, more diffusion processes occur. This leads to a finer grained eutectic phase with increasing holding time (compare figures 5a, b and c). The whole niobium foil has reacted with the base material to the eutectic after a dwell time of only 1 s. Furthermore, an increasing dwell time leads to a growth of proeutectic dendrites. The average thickness of this phase increases from approx. 10 µm (dwell time 1 s) to 15 µm (1 min) to 25 µm (34 min).

For tensile testing one specimen was brazed at 1180 °C for 6 min. The tensile strength of the specimen is 1022 MPa. The fractured surface of this specimen is shown in figures 5d and e. The failure occurred in the eutectic and partly in the proeutectic phase. Though, for validation of the tensile strength and the failure mechanism more tensile tests are necessary.

Figure 4. SEM image with element analysis of a NiTi brazed joint with pure niobium (50µm) as brazing filler metal. Brazing parameter: 1180 °C, 6 min.

Figure 5. a-d) SEM images of NiTi joints with different holding times (brazing temperature 1180 °C), d) SEM Images of the fractured surface of a tensile specimen, e) cross section of the fractured surface.

4. Conclusions

It could be proven that solid solution annealing between 800-1180 °C with a subsequent shape-set heat treatment of 510 °C in a common vacuum furnace preserved a high level of pseudoelasticity for NiTi-wires. A regular straight annealed condition as well as a cold-formed condition (φ=40,7 %) were used in the original state. It was found that there was a largely R-phase free conversion for the regular
condition after heat treatment leading to tension plateau of 285 MPa for the wires annealed to 1180 °C. This was 35 MPa higher than for the wires which were annealed at only 800 °C. In contrast to this, there was a non-linear stress-strain behavior present for the heat-treated wires which were in the cold-formed state before. This suggests the presence of the R-Phase which indicates an incomplete dissolution of dislocations. Nevertheless, it could be proven for the first time that these wires offer a high degree of pseudoelasticity. The level of the tension plateau was 305 MPa for a solution annealing temperature of 1180 °C whereas it was just 200 MPa for the solution annealed wires at 800 °C.

Furthermore, it was shown that vacuum brazing of NiTi using pure niobium led to high quality and sound joints. In accordance to the literature, the microstructure consisted of a quasieutectic NiTi-Nb phase and proeutectic phase which was increased with the dwell time. A tensile strength of 1022 MPa was achieved for a brazed sample with a dwell time of six minutes. The fracture was within the eutectic phase so that it will be interesting to investigate the effect of higher dwell times on the tensile strength and the pseudoelasticity in future in detail.

In future, the project will investigate the effect of the heat treatment process on NiTi materials in detail and systematically for the regular pseudoelastic condition as well as for the cold-formed condition. In this regard, the grain growth, the microstructure and the pseudoelastic behavior are of main interest. As a consequence of these findings, limiting process variables for brazing with niobium are derived. In addition to brazing with Nb, NbZr1, Cu, and AuCu65 are promising brazing alloys to achieve a partial pseudoelastic behavior within a NiTi/NiTi-joint.

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