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Microstructural Analysis of vacuum-brazed NiTi-Stainless Steel using Cu and Nb filler

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Abstract:

Nickel-titanium (NiTi) alloys exhibit a pronounced shape memory effect, which is of particular importance for applications in medical devices and electronic actuators. As the shape memory effect is dependent of the microstructure of the base material, which can be degraded by excessive heat exposure, a precise thermal control as well as limited heat input during the joining process have to be ensured in order to preserve these functional properties. In this regard, joining at comparably low temperatures is favorable for the manufacture of NiTi-AISI304 joints. In contrast to welding, brazing operates at lower temperatures and therefore offers significant potential to maintain the shape memory characteristics of NiTi. Hence, this study investigates the dissimilar joining of NiTi and stainless steel (AISI304), with a focus on microstructural characterization. Varying filler metals, including copper- and niobium-based alloys, were employed to investigate the influence of both holding time and filler metal composition on the microstructural evolution within the brazing seam. In all cases, sound metallurgical bonding was achieved. However, the formation of intermetallic phases in the brazing seam was observed. These phases are expected to adversely affect the functional properties of the NiTi alloy, particularly its shape memory behavior. The results underline the strong influence of process parameters on phase formation and provide a basis for optimizing brazing conditions for NiTi–steel joints.

Introduction:

Nickel-titanium (NiTi) belongs to the class of shape memory alloys (SMAs). These materials are characterized by their ability to recover their original shape after deformation. This behavior is based on the reversible, diffusion-free crystal phase transformation between the metastable austenitic phase and the martensitic phase [1;2;3]. One part of the shape memory effect is pseudoelasticity, in which the material undergoes large reversible strains under mechanical loading due to a stress-induced transformation from austenite to martensite and the subsequent reverse transformation upon unloading [4].

Owing to these special property, the shape memory, NiTi is widely used in various technological fields. In particular, the biomedical sector represents one of the most important areas of application. Devices such as stents, medical guidewires and orthodontic components benefit from the high recoverable strain, good corrosion resistance and the high biocompatibility of NiTi [5;1;6;7;8].

The functional behavior of NiTi is highly sensitive to its chemical composition. In particular, the ratio of nickel to titanium has an influence on the transformation temperatures and therefore on the resulting shape memory and pseudoelastic properties. Typically, the nickel content must remain within a narrow range of approx. 49 - 52 at.% Ni, with the balance being titanium [4]. Even minor deviations from this composition can alter the transformation behavior and impair the functional properties [9]. Due to the temperature-dependent, diffusion-free transformation from austenite to martensite, joining NiTi is challenging. During welding processes, the high thermal input can lead to evaporation of alloying elements and local compositional changes within the joining zone. As a result, the functional properties of NiTi, especially its pseudoelastic

behavior, may deteriorate or even be completely lost [4;10]. Therefore, laser welding is widely used because it enables the production of high-strength joints while generating only a small heat-affected zone. [10;11]. However, this technique is mainly limited to wire-to-wire or butt joint configurations, and the formation of precipitates in the weld metal reduces the maximum attainable strain [12].

Previous studies have indicated that vacuum brazing represents a promising alternative for joining NiTi components [13-17]. Compared to welding process, brazing operates at lower temperature and avoids melting of the base material. Experimental investigations have shown that with appropriate control of the brazing temperature and cooling conditions, the pseudoelastic behavior of NiTi can largely be preserved [15;17]. In particular, it was demonstrated that slow cooling promotes the transformation of stress-induced martensite to austenite, resulting in the loss of pseudoelasticity. In contrast rapid cooling using an argon overpressure of 2 bar successfully restored the pseudoelastic response [17]. In previous publications could be proven, that sound joints were obtained, when brazing NiTi to NiTi using pure copper resp. Au65Cu as filler materials [14].

Joining NiTi to dissimilar materials is particular interest, as it enables the investigation of how the shape memory properties are affected by the joining process and the interaction between different materials. Austenitic stainless steel, AISI304, represents a promising joining partner due to its corrosion resistance, good biocompatibility, widespread availability and cost-effextiveness [2]. Previous studies have also investigated the joining of NiTi to stainless steels using processes such as laser welding and infrared brazing, demonstrating the general feasibility of such dissimilar joints. However, these connections are often critically assessed due to the formation of brittle intermetallic phases. Particularly Fe-Ti compounds, which can reduce the mechanical performance of the joint [18-24]. Investigations on NiTi to AISI316L with Ag-based filler metals revealed the formation of CuNiTi phases at the NiTi reaction zone and Fe_2Ti as well as (Fe,Ni)Ti reaction layers at the steel interface, which tend to exhibit brittle fracture behavior. The resulting cracks frequently observed within the Fe-Ti reaction zone during mechanical testing, identifying this region as a critical point of the joint. Furthermore, the mechanical strength of the brazed connections was found to depend on the process temperature and the holding time. It was shown, that lower brazing temperatures combined with longer holding times can improve joint quality and mechanical performance [24].

Experimental and materials:

A sample geometry of 10 mm x 10 mm x 2 mm was used. The NiTi samples (Ingplus Medical GmbH, Inc.) had a chemical composition of 50,8 - 51,1 at.% Ni and 48,9 - 49,2 at.% Ti. The AISI304 samples (ESC Burg) consisted of 17,5 - 19,5 at.% Cr, 6-10,5 at.% Ni, max. 0.07 at.% C, max. 2 at.% Mn, max. 1 at.% Si, max. 0.045 at.% P, max. 0.03 at.% S, and max. 0.1 at.% N. Niobium (Sondermetalle e.K.) and copper (LOT-TEK) with a purity of 99,99% were used as filler metals. The thickness of the brazing foils was 50 µm. The brazing foil was placed between the NiTi and AISI304 and the samples were placed in the vacuum furnace (Schmetz EU80/1H 30x45x30) and a weight of approx. 28 g was placed on top of each sample. For the experiments conducted in this study, a vacuum of at least 10^{-5} mbar was realized. Temperature control of the furnace processes was ensured using a thermocouple. During the brazing process, a heating rate of 10 K/min was applied up to homogenization temperature of 200°C (see Fig. 1). To ensure a homogeneous temperature distribution within the furnace chamber and the samples, this temperature was held for 5 minutes. Subsequently, the furnace chamber was heated to the brazing temperature at a heating rate of 50 K/min. For niobium a brazing

temperature of 1180°C was chosen, which is approx. 10 °C above the eutectic temperature of the Nb-NiTi system at 1170°C [25]. For copper, a brazing temperature of 1095°C was selected, which is about 10°C above the melting temperature of copper (1084,9°C) [18]. The brazing temperature was maintained for holding times of either 5 minutes or 30 minutes.

After the holding time was reached, the furnace chamber was cooled using an argon cooling gas flow at 2 bar with a cooling fan speed of 3000 rpm. The high cooling rate was applied in order to suppress undesirable phase transformations [15;17]. To avoid the formation of the R-Phase, which may occur during exposure to temperatures between 300°C to 500°C, a part of the specimens underwent a post heat treatment. During this shape annealing, the samples were heated to 510°C, held at this temperature for 5 minutes with a subsequent fast cooling.

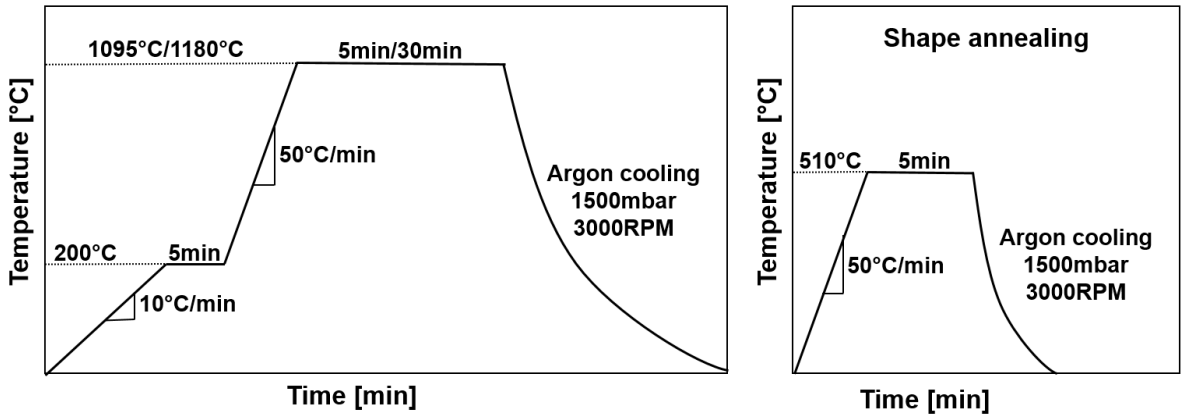


Figure 1: Temperature profile of the brazing cycles

Results and discussion:

Microstructure of NiTi -AISI304 joints with Cu filler metal

Figure 2 shows the cross-sections of the brazed samples. For All specimens, good bonding was achieved, with no defects, pores, or voids observed at the interfaces. The samples are arranged with the stainless steel on top, the brazing seam (with copper filler metal) in the middle and the NiTi at the bottom. The upper row shows samples with a holding time of 5 minutes, while the lower row shows samples held for 30 minutes. The right-hand column shows samples that underwent an additional heat treatment (shape annealing) after brazing. For the specimens with a holding time of 30 minutes, the post heat treatment could not be performed, since softening of NiTi specimens occurred at this condition.

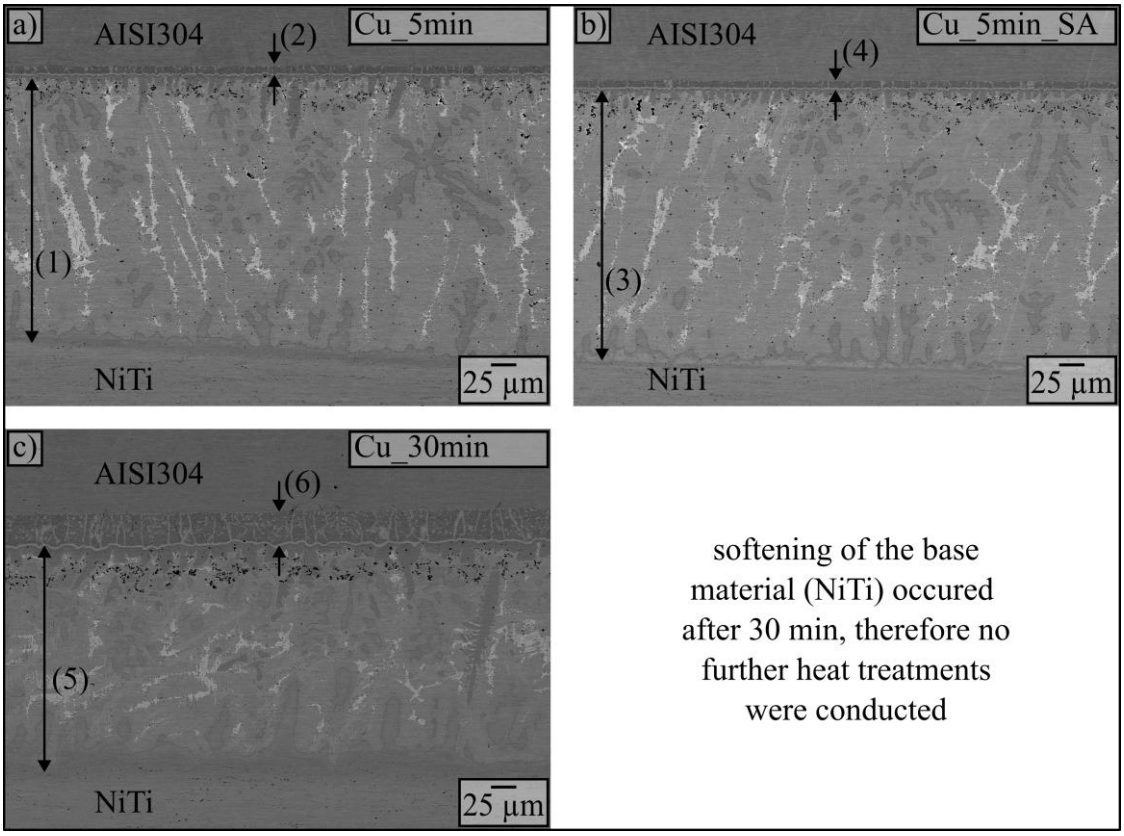


Figure 2:Overview cross-sections of the brazed samples with Cu filler metal brazed for 5 resp. 30 min with and without post heat treatment

In all samples, a dark reaction zone is visible at the interface to the stainless steel, with a continuous bright phase band located in the center of this zone. Additionally, dark spots can be observed on the steel side. The thickness of the reaction zone increases with increasing holding time: comparing Figure 2A (9 µm) with Figure 2 (30 µm) shows a clear thickening of the reaction zone, accompanied by a slight reduction in the brazing width (from 280 µm to 220 µm). On the NiTi side, a dark phase is visible, which is consistent with the pro-eutectic phase reported in the literature. Furthermore, bright phases can be observed throughout the entire brazing seam.

Overall, the microstructure does not differ substantially with respect to holding time or heat treatment. For this reason, the following discussion focuses on the 30-minutes sample, as the phases are most pronounced in this condition. To gain a more detailed understanding of the microstructure, EDS mappings were performed (Figure 3). The results reveal a chromium enrichment on the AISI304 side, indicating a possible chromium depletion within the stainless steel. Furthermore, the previously identified reaction zone is confirmed to consist of iron-chromium-titanium-rich phases. Titanium-rich phases are additionally observed at both the steel/braze metal and braze metal/NiTi side. The dark spots neat the steel side are most likely titanium oxides, consistent with findings from previous studies [15].

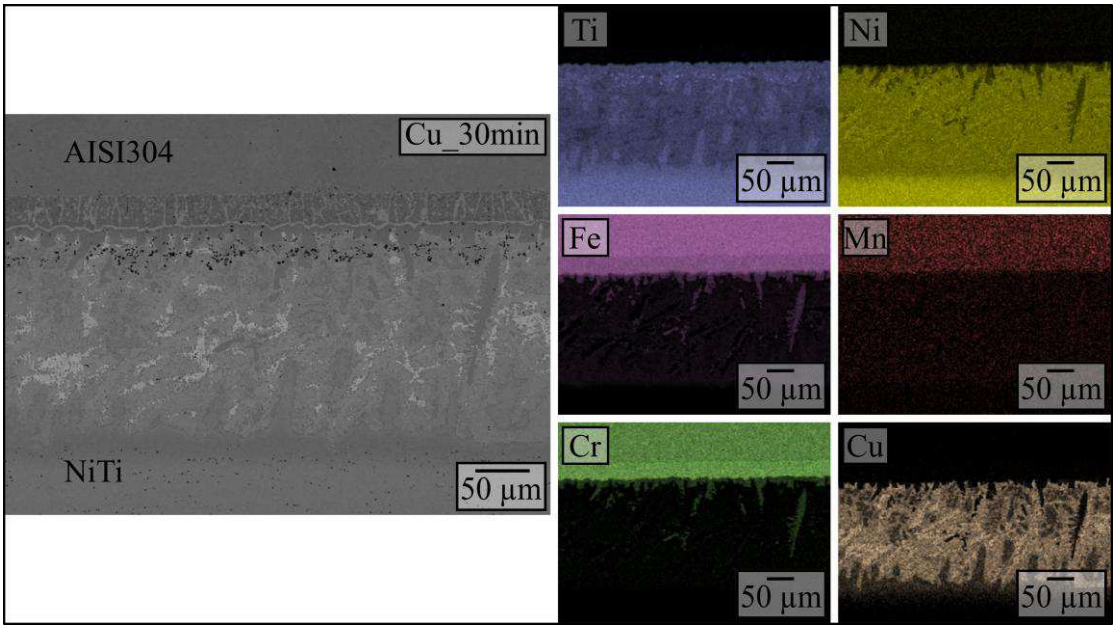


Figure 3: EDS-Mapping of a brazed sample using Cu filler metal and a dwell time of 30 min without post heat treatment

A closer examination of the EDS-analysis is provided in Figure 4 and Table 1. Points 1 and 2, located in the reaction zone near the brazing seam, show chromium enrichment, which further supports the assumption of chromium depletion in the stainless steel. Near the AISI304 side, a dark grey phase (point 3) can be identified as an FeTi intermetallic phase. According to the literature, such phases indicate the presence of brittle intermetallics, which have a negative impact on pseudoelasticity. The brighter phase (point 4) corresponds to a NiCuTi-rich phase. The dark grey phase neat the NiTi (point 6) is NiTi-rich and indicates the presence of the pro-eutectic. However, this phase also contains iron, which diffused from the steel. At point 7, a titanium depletion is observed within the NiTi base material. Since NiTi is a binary alloy, a reduction in titanium content corresponds directly to an increase in the relative nickel content. According to the literature, the nickel content must remain between 49 and 52 at.% to preserve the pseudoelastic behavior [4]. However, at point 7, a nickel content of 57 at.% was measured, confirming that the titanium depletion has shifted the local composition well beyond this critical range. This elevated nickel content may contribute to the observed softening of the base material, as increasing nickel content is known to significantly lower the martensitic transformation temperature of NiTi. A compositional shift of only 1 at.% can change the transformation temperature by up to 100 K [26]. As a result of the local Ni enrichment, the transformation temperature at point 7 is likely shifted well below the testing temperature, causing the material to remain in or transform into the softer, lower-strength austenitic phase rather than exhibiting its characteristic pseudoelastic response.

Additionally, hardness testing was performed. The reaction zone exhibited a hardness of 453.9 ± 43.1 HV0.01, indicating a moderately hardened region on the steel side. In contrast, the dark grey phase, identified as an FeTiNi-rich phase, showed a considerably higher hardness of 845.6 ± 7.9 HV0.01. Given its high hardness, this intermetallic phase is expected to have a detrimental effect on the pseudoelastic behavior of the joint.

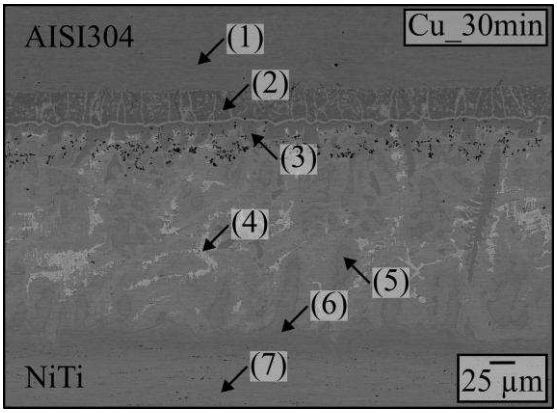


Figure 4: Microstructure of the NiTi-AISI304 samples (with Cu filler metal) with a 30 min holding time

Table 1: Chemical composition of the NiTi-AISI304 samples brazed with Cu filler metal at a holding time of 30 min

	Ti (at.%)	**Ni** (at.%)	**Fe** (at.%)	**Cr** (at.%)	**Si** (at.%)	**Mn** (at.%)	**Cu** (at.%)
(1)	-	8,17	71,53	18,24	0,38	1,68	-
(2)	7,01	6,71	62,29	21,12	0,17	1,52	1,19
(3)	28,33	17,02	41,62	9,68	0,95	0,56	1,84
(4)	21,36	46,03	6,41	0,85	-	-	25,35
(5)	33,54	39,68	13,27	2,76	-	0,57	10,18
(6)	36,40	39,97	13,45	2,23	-	-	7,95
(7)	42,98	57,02	-	-	-	-	-

The microstructural and compositional analysis of the copper-brazed NiTi/stainless steel joint reveals a well-bonded interface, free of defects, pores, or voids. A reaction zone forms at the steel side, consisting of iron-chromium-titanium-rich phases, the thickness of which increases with holding time. This reaction zone is associated with a moderate hardness increase, while the FeTi-rich intermetallic phase in the brazing seam exhibits considerably higher hardness, likely contributing to embrittlement at the interface. On the NiTi side, a pro-eutectic phase forms, accompanied by localized titanium depletion and a corresponding nickel enrichment of up to 57 at.% at selected points. This compositional shift is expected to lower the local martensitic transformation temperature, which may explain the softening of the NiTi base material observed for the 30-minute holding time. Overall, the results indicate that both the reaction zone thickness and the degree of compositional shift in the NiTi base material increase with holding time, with direct implications for the pseudoelastic performance of the joint.

Microstructure of NiTi -AISI304 joints with Nb filler metal

Figure 5 shows an overview of the microstructure of the NiTi-AISI304 joints brazed using Nb as filler metal. The upper row shows samples with a holding time of 5 minutes, while the lower row shows samples with a holding time of 30 minutes. The right-hand column shows samples that additionally underwent a post-braze heat treatment (shape annealing). As already observed for the Cu filler metal, a reaction zone is visible on the steel side. Dark phases are present at both interfaces, i.e., steel/braze and braze/NiTi, and are attributed to pro-eutectic

phases, as already known from the literature. In addition, a eutectic reaction is visible within the brazing seam. A comparison of Figure 5A and Figure 5C reveals an increase in reaction zone thickness with increasing holding time (20 µm for 5-minutes sample vs. 45 µm for the 30-minute sample), accompanied by a corresponding reduction in the brazing seam width (from 215 µm to 145 µm). This trend is consistent between the samples with and without shape annealing. Furthermore, a continuous phase is observed between the reaction zone and the pro-eutectic phase, which is more pronounced in the 30-minutes samples. For this reason, and because all phases are most distinctly developed in this sample, the 30-minute sample is analyzed in more detail in the following section.

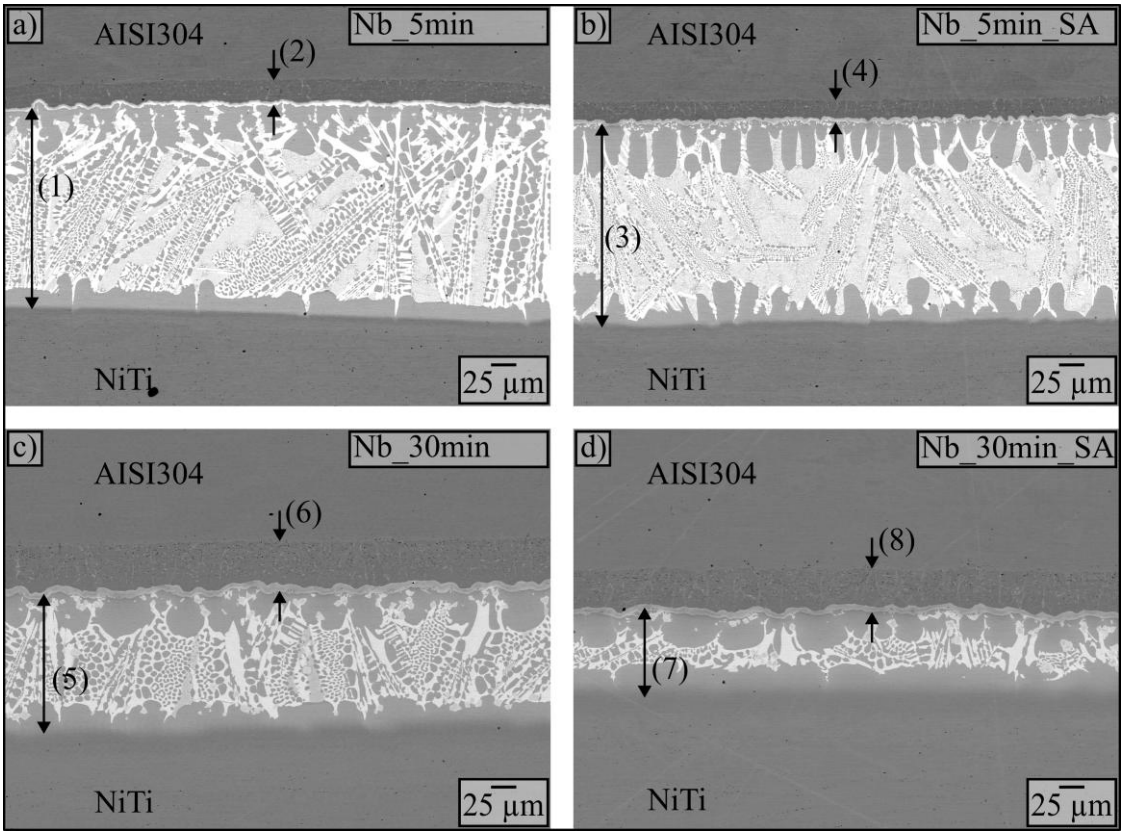


Figure 5: Overview of cross-sections of the samples brazed with Nb filler metal for 5 resp. 30 min with and without post heat treatment

The EDS mapping (Figure 6) reveals a chromium enrichment near the reaction zone, indicating a possible chromium depletion in the steel. The reaction zone itself appears to be FeCr-rich. Furthermore, the regions previously assumed to be pro-eutectic phase are shown to actually consist of NiTi-rich phases. A eutectic structure between NiTi and Nb is visible within the brazing seam, and the bright phases in the brazing seam are identified as Nb-rich. The continuous phase band between the reaction zone and the brazing seam appears to be FeNbTi-rich.

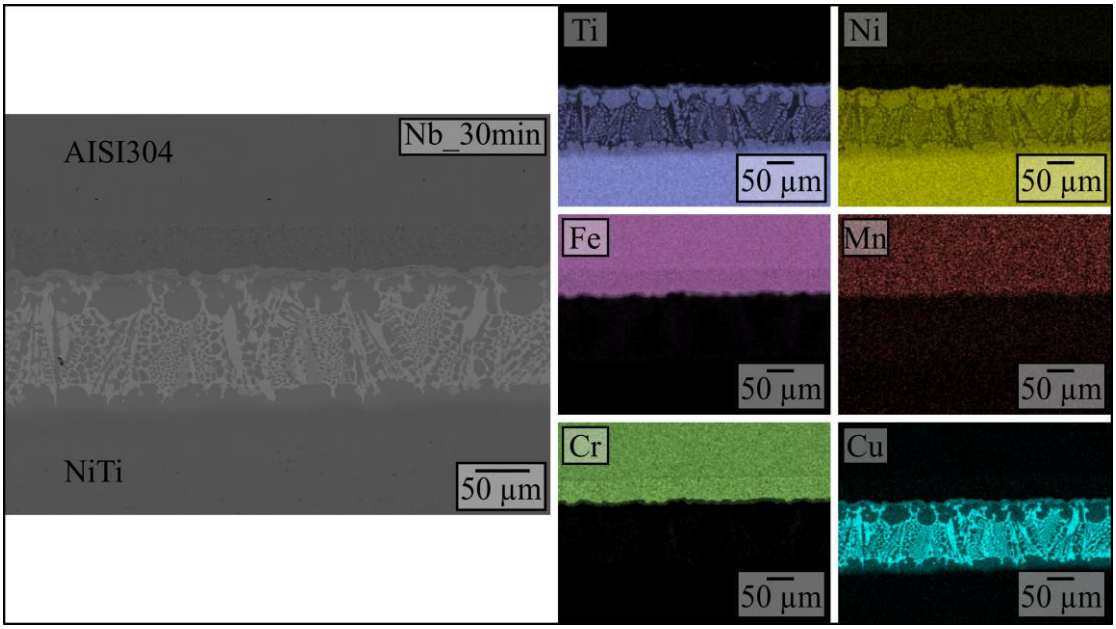


Figure 6: EDS-Mapping of a sample brazed using Nb filler metal f0r 30 min without post heat treatment

A closer examination in Figure 7 confirms the FeNbTi-rich phase (point 3 in Table 2). Additionally, a comparison of points 1 and 2 confirms chromium enrichment within the reaction zone. The NiTiNb phase observed at both sides (point 4 and point 6) is indicative of a pro-eutectic phase, which may impair pseudoelastic behavior. A titanium depletion is observed in the NiTi base material, although to a minor extent than in the Cu-brazed joints. Hardness testing further revealed differences between the individual phases within the brazing seam. The pro-eutectic phases exhibited a comparatively lower hardness of 416,28 ± 12,37 HV0.01, while the continuous FeNbTi reaction layer at the steel side (point 3) showed a hardness of 1018,39 ± 40,15 HV0.01, representing an additional brittle phase.

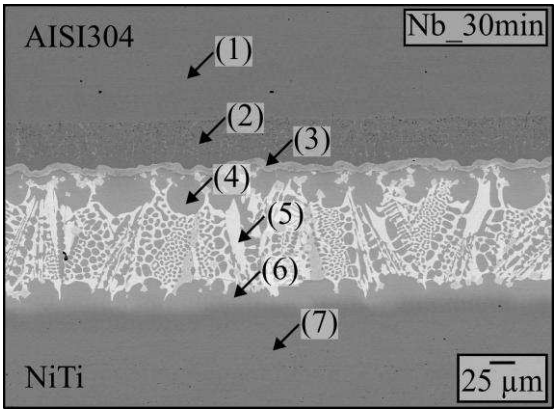


Figure 7: Microstructure of the NiTi-AISI304 samples (with Nb filler metal) with a 30 min holding time

Table 2: Chemical composition of the NiTi-AISI304 samples brazed with Nb filler metal at a holding time of 30 min

	Ti (at.%)	**Ni** (at.%)	**Fe** (at.%)	**Cr** (at.%)	**Si** (at.%)	**Mn** (at.%)	**Nb** (at.%)
(1)	-	8,23	71,36	18,29	0,39	1,72	-
(2)	1,36	5,74	69,32	21,86	0,15	1,57	-

(3)	13,43	7,72	48,26	8,23	1,28	1,01	20,06
(4)	33,13	41,67	7,33	0,58	-	-	17,29
(5)	13,66	25,90	8,48	2,79	-	-	49,17
(6)	32,98	42,71	6,45	0,57	-	-	17,28
(7)	44,69	55,31	-	-	-	-	-

A closer look reveals square-shaped phases distributed throughout the brazing seam (Figure 8). Hardness testing of these phases yielded an extremely high value of 1282.9 ± 149.2 HV0.01, indicating pronounced brittleness.

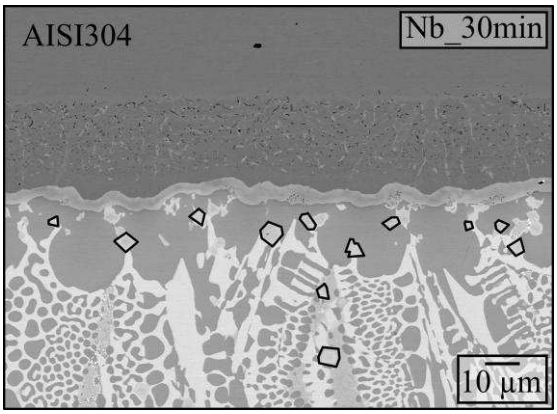


Figure 8: Brittle phases in the NiTi-AISI304 samples (Nb filler metal) with 30 min holding time

The microstructural and compositional analysis of the Nb-brazed NiTi/stainless steel joint reveals a well-bonded interface, free of defects, pores, or voids. A reaction zone forms at the steel side, consisting of FeCr-rich phases together with a continuous FeNbTi-rich phase band, the thickness of which increases with holding time while the brazing seam width decreases accordingly. Within the brazing seam, a eutectic structure between NiTi and Nb is present, along with Nb-rich bright phases and pro-eutectic NiTiNb-rich phases at both interfaces. Hardness testing revealed pronounced differences between the individual phases: while the pro-eutectic phases showed comparatively lower hardness, the square-shaped phases distributed throughout the brazing seam exhibited an extremely high hardness of 1282.9 ± 149.2 HV0.01, indicating pronounced brittleness. A titanium depletion is also observed in the NiTi base material, suggesting a local shift in the martensitic transformation temperature. Overall, the results indicate that both the reaction zone thickness and the extent of brittle phase formation increase with holding time, with the presence of hard, brittle intermetallic phases likely having a detrimental effect on the pseudoelastic performance of the joint.

Conclusion:

Sound, defect-free joints between NiTi and AISI 304 stainless steel were successfully achieved using both Cu and Nb as filler metals. In both cases, a chromium-enriched reaction zone formed at the steel interface, indicating potential local chromium depletion in the AISI 304, while the post-braze heat treatment showed no significant influence on the resulting microstructure. However, the two filler metals led to distinctly different phase formations within the brazing seam, with corresponding implications for the pseudoelastic performance of the joint.

1) Shape annealing after brazing had no significant influence on the observed microstructure.

2) Reaction zone thickness increased with increasing holding time for both filler metals, accompanied by a corresponding reduction in brazing seam width.
3) In both cases, a chromium-enriched reaction zone formed at the steel interface, indicating a potential for chromium depletion in the AISI 304 base material.
4) Cu-brazed joints: formation of FeTi-based intermetallic phases, which may impair pseudoelastic behavior
5) Cu-brazed joints: titanium depletion in NiTi due to preferential Ti diffusion into Cu, resulting in softening of the base material.
Nb-brazed joints: titanium depletion in NiTi was also observed, though less pronounced than in Cu-brazed joints
6) Nb-brazed joints: formation of continuous NbTiNi and FeNbTi intermetallic phases with very high hardness

Further work will investigate the influence of diffusion barriers consisting of tungsten. These investigations aim at suppressing elemental interdiffusion leading to a reduction of the formation of brittle intermetallic phases.

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Competing interests

The authors have no competing interests to declare that are relevant to the content of this article.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Author contributions

Alexa Nebel and Lukas Wojarski contributed to the study conception and design. Material preparation, experiments, data collection and analysis were performed by Moritz Ridder and Alexa Nebel. The first draft of the manuscript was written by Alexa Nebel supervised by Lukas Wojarski, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript. Lukas Wojarski as project leader is responsible for the scientific approach.

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