Supplementary Material Document for   
Integration of AlN Piezoelectric Thin-Films on Ultra-Low Fatigue TiNiCu Shape Memory Alloys

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**I: Analytical Calculation and Experimental Demonstration of Resonant Frequency**

**A. Theory:**

The primary goal of the analytical calculations performed in this study are to quantify the contribution of the Si thickness (tSi) and NiTi thickness (tNiTi) on the change in mechanical resonant frequency (Δf). The maximum shift is achieved by thermally inducing a reversible phase transformation in the shape memory alloy from martensite to austenite. The resonant frequency of a cantilever-based magnetoelectric (ME) / shape memory alloy (NiTi) / silicon (Si) composites is modeled as the first bending mode of a freestanding cantilever composed of three mechanically coupled thin-film layers: ME / NiTi / Si. The theory behind the calculation of resonant frequency of a two-layer thin-film cantilever beam is well known.1–3 The theory and Matlab code presented in *Zannon et al*.1 is employed to expand the calculation to three-layers in order to investigate ME/ NiTi/ Si composites.

The Si and NiTi functional layers will dominate the elastic properties of the vibration due to their large film thickness; therefore, the mechanical contributions of the adhesion and electrode layers can be neglected. To simplify the calculations, the magnetoelectric layer uses the mechanical properties of the piezoelectric layer (AlN) and magnetostrictive layer (FeCoSiB) mechanically coupled together. In all calculations the ME thickness is assumed tME = 4 μm to match the functional layer thickness (i.e tAlN = 2 μm, tFeCoSiB = 2 μm) of the fabricated composites experimentally analyzed in this manuscript. Taking a similar approach to *Röbisch et al.*3 the combined mechanical properties of AlN and FeCoSiB are used to calculate the Young’s Modulus (EME = 230 GPa) and density (ρME = 5275 kg/m3) of the ME layer as shown in Equation S1 and Equation S2, respectively. These properties are estimated by using the thickness weighted average of the AlN and FeCoSiB functional layers.

(Equation S1)

(Equation S2)

As described in *Zannon et al.,*1 the resonant frequency of multi-layer composites in cantilever structures can be described in terms of the moment of inertia, and elastic modulus of the individual layers. As additional thin-film materials layers are added to the composite stack, it will induce a shift in the center of mass ( and inertia (of the beam layers, as shown in Equations S3 – S6.

(Equation S3)

(Equation S4)

(Equation S5)

(Equation S6)

Equation S7 demonstrates the resonant frequency is dependent on the geometrical dimensions of the cantilever: width (*w*), length (*L*), and thickness as well as the Young’s modulus (*EX*), density , moment of inertia (*Ix*), and in-plane surface area (*Ax*) of each material layer ( = ME, NiTi, Si or 1, 2, 3*).* Here *A* is the in-plane surface area: (i.e short axis cross section) of the cantilever. λ is an integration constant determined by the boundary conditions (*λ* = 1.875) for the first bending mode of a cantilever beam.4 The same cantilever geometry (w = 2.5 mm x L = 25 mm) is held consistent in all calculations.

(7)

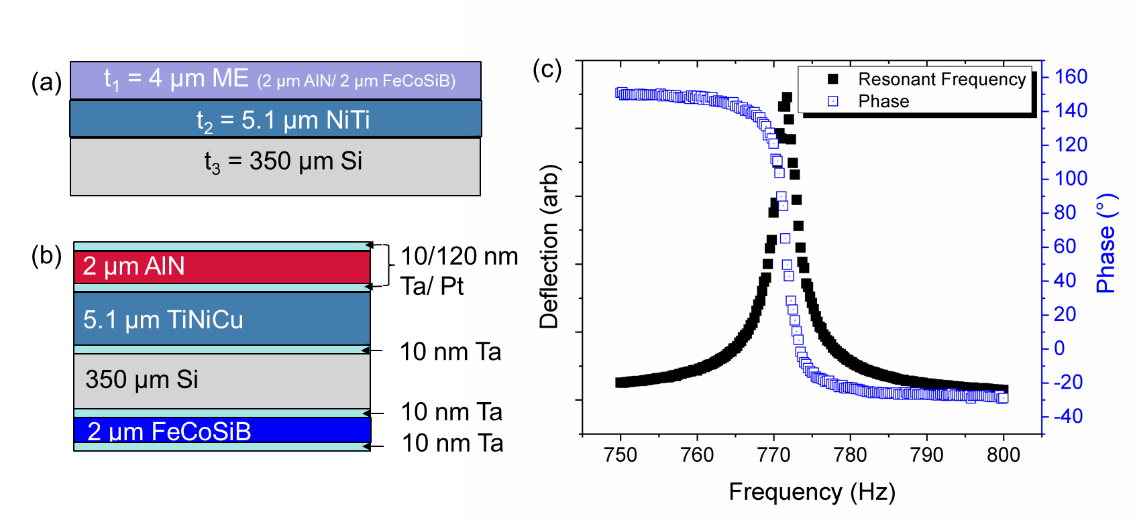
Since it is possible to gradually and reversibly change the Young’s modulus of NiTi by inducing a partial phase transformation, a gradual change in the resonant frequency can be obtained. The largest Δf will occur when there is a complete phase transformation from the low-temperature martensite phase (*ENiTi,martensite* = 41 GPa) to the high-temperature austenite phase (*ENiTi,austenite* = 83 GPa). Equation S8 demonstrates the expression used to calculate the maximum

(8)

**B. Experimental Verification of the Model:**

The Matlab code output of the first bending mode of a 4 μm ME / 5.1 μm NiTi / 350 μm Si composite predicts a maximum resonant frequency shift (Δf = 0.5%) from 768 Hz to 772 Hz. The mechanical properties of binary NiTi (EMartensite = 41 GPa, EAustenite = 83 GPa, ρ = 6450 kg/m3)5 are well studied and like that of TiNiCu (EAustenite = 78 GPa, ρ = 6500 kg/m3).6 Therefore, we expect a maximum deflection (i.e first bending mode resonant frequency) of the fabricated 2 μm AlN/ 5.1 μm TiNiCu/ 350 μm Si/ 2 μm FeCoSiB cantilever composite to fall within this range. Figure S1(a) demonstrates the cross-section of the ME/ SMA/ Si composites investigated through analytical calculations. This is the analytical equivalent composite to the fabricated experimental composite, shown in Figure S2(b).

The experimental resonant frequency was obtained using an optical read-out deflection set-up, similar to the one described elsewhere.7 The end 1 mm of the fabricated 2.5 mm x 26 mm cantilever ME/ SMA/ Si composite was fixed onto a holder (freestanding length 25 mm). A time varying magnetic field, having a nonzero DC component matching the working point of the ME composite, is used to excite the magnetostrictive FeCoSiB layer, which is mechanically coupled to the piezoelectric layer of AlN, thus inducing the composite magnetoelectric effect. When this composite is sent into mechanical resonance, a vast increase in deflection amplitude is observed. Figure S2(c) shows the obtained experimental mechanical resonance frequency at 771 Hz with a corresponding phase change also at 771 Hz which indicates a maximum deflection. This result is in excellent agreement with the analytical calculation, using the mechanical properties of binary NiTi, which predicts a resonant frequency of 768-772 Hz.

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**Figure S1:** (a)Cross-section of the modeled ME / NiTi / Si composite used in analytical frequency calculations. (b) Cross-section of the fabricated AlN / TiNiCu/ Si/ FeCoSiB composite. (c) Resonant frequency and phase measurement of AlN / TiNiCu/ Si/ FeCoSiB composite taken by optical beam deflection while magnetically excited. An experimental resonant frequency of 771 Hz was obtained.

**II: Fabrication Parameters**

The texture and smoothness of the underlying substrate will strongly influence the piezoelectric performance of AlN thin-films.8 To remove any impurities from the substrates, all Si substrates were first cleaned in a series of acetone, isopropyl alcohol, and water ultra-sonic baths, followed by heating on a hot-plate to 100 °C. All layers were sputtered using a Von Ardenne (CS 730S) magnetron cluster system, capable of DC, RF, and Pulsed DC sputtering. Prior to all depositions, a free-sputter for a minimum of 5 minutes took place on all targets to ensure the removal of impurities. Prior to deposition of the adhesion layer (Ta) and piezoelectric layer (AlN), the first few nm of the sample were plasma etched in Ar atmosphere.

Low-temperature magnetron sputtering of AlN from an aluminum (Al) target in a reactive environment is a well-established process. Here, we follow the recipe established by *Yarar et. al..*9AlN is deposited through a pulsed DC reactive sputtering process using ultra-pure (5N) N2 gas and a high purity Al-target (99.999%). The Al target was first conditioned via DC free-sputtering with Argon gas (Ar) on an empty carrier plate for 15 minutes. Afterward, AlN was also conditioned for 15 minutes on the carrier plate via pulsed DC reactive sputtering, powered by a Pinnacle Plus+ 10kW (Advanced Energy) asymmetric bipolar pulsed DC power supply sputtering operating at a frequency of 250 kHz with 1.6 µs reverse time. After these conditioning steps took place, ~2 µm of AlN was sputtered under the same conditions on the sample for 42 minutes. Finally, ~2 µm FeCoSiB is sandwiched between two layers of Ta (adhesion and protection layers) by means of RF sputtering on the backside of the wafer. The sputtering parameters used for depositions of all materials layers in the fabricated composite are shown in Table SI.

TABLE SI. Sputter deposition and fabrication processing parameters of each material in the AlN / TiNiCu/ Si/ FeCoSiB composites

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Material Layer | Sputter Target [diameter] | Power | | Sputter Pressure | | Ar / N2 flow | Time | | Film thickness | | | Additional Processing Notes | |
| Standard DC Sputtering: | |  | |  | |  |  | |  | | |  | |
| Pre-etch | ----- | 100 W | | 5E-3 mbar | | 30 Ar | 1 min | | ---- | | | Removes first few nm | |
| Adhesion | Ta [8in] | 200 W | | 4E-3 mbar | | 30 Ar | 1 min | | 20 nm | | |  | |
| SMA | Ti50Ni35Cu15 [8 in] | 600 W | | 2E-3 mbar | | 25 Ar | 66 min | | 5.1 μm | | |  | |
| Adhesion | Ta [8 in] | 200 W | | 4E-3 mbar | | 30 Ar | 0.5 min | | 10 nm | | |  | |
| Bottom Electrode | Pt [4 in] | 100 W | | 8E-3 mbar | | 25 Ar | 1.5 min | | 100 nm | | |  | |
| Free Sputter | Al [8 in] | 200 W | | 6E-3 mbar | | 30 Ar | 15 min | | ---------- | | |  | |
| Pulsed DC Reactive Sputtering: (after Al and AlN conditioning steps) | | | | | | | | | |
| Piezoelectric (AlN) | Al [8 in] | 1500 W pDC | | 1.7E-3 mbar | | 10 N2 | 42 min | | 2 μm | | | Time = 1.6 μs,  Frequency = 250 kHz | |
| Standard DC Sputtering: | |  | |  | |  |  | |  | | |  | |
|  | |  | |  | |  |  | |  | | |  | |
| Adhesion | Ta [8 in] | 200 W | | 4E-3 mbar | | 30 Ar | 0.5 min | | 10 nm | | |  | |
| Top Electrode | Pt [4 in] | 100 W | | 8E-3 mbar | | 25 Ar | 1.5 min | | 100 nm | | |  | |
| RF Sputtering: | |  | |  | |  |  | |  | | |  | |
|  | |  | |  | |  |  | |  | | |  | |
| Adhesion | Ta [8 in] | 400 W | | 4E-3 mbar | | 30 Ar | 17 seconds | | 10 nm | | |  | |
| Magnetostrictive (FeCoSiB) | (Fe90Co10)78Si12B10  [8 in] | 200 W | | 6E-3 mbar | | 40 Ar | 230 min | | 2 μm | | |  | |
| Protection | Ta [8 in] | 400 W | | 4E-3 mbar | | 30 Ar | 17 seconds | | 10 nm | | |  | |
|  |  |  |  | |  | |  |  | | |  | |

**III: Shape Memory Alloy Characterization**

Temperature dependent XRD (Anton-Paar DHS 1100) measurements on a fabricated AlN/TiNiCu/ Si composite were performed to observe a fully reversible phase transformation. between 30 °C to 100 °C to ensure a full reversible phase transformation from martensite to austenite and again at 30 °C to evaluate if TiNiCu fully returns to martensite. Figure S2(a) demonstrates upon heating to 100 °C in 10°C steps, no observable change in the (002) AlN peak position (2θ = ~36°) or intensity is detected. This indicates that the phase transformation does not affect the structural properties of the piezoelectric material. Figures S2(b) – S2(d) show selected temperature dependent XRD peaks confirming a full phase transformation from the B19 (martensite orthorhombic structure, space group PMMB #51) to the B2 (austenite simple cubic structure) upon heating to temperatures above 70 °C. The expected peak position of the martensite (M) and austenite (A) peaks obtained from the TOPAS software are listed in Table SII. These transformation temperatures are in good agreement with the DSC data performed on a the 20 μm thick freestanding TiNiCu films shown in Figure 3 in the main manuscript.

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**Figure S2:** Temperature dependent XRD measurements on a AlN/ TiNiCu/ Si composite (Sample 3 after annealing) heated from 30 °C to 100 °C and back to 30 °C. (a) (002) AlN peak demonstrating no change upon heating to 100 °C. (b) – (d) Shows the select temperature dependent diffractograms demonstrating the phase transformation from martensite (M) to austenite (A) occurs between 60 – 70 °C in TiNiCu films after a heat treatment to 700 °C for 15 minutes.

TABLE SII: Select data-base x-ray diffraction peak positions for TiNiCu martensite and austenite phases

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Martensite Peaks (B19) | |  |  | Austenite Peaks (B2) | |
| Angle | Plane | Angle | Plane | Angle | Plane |
| 42.44° | (020) | 42.64° | (111) | 42.09° | (110) |
| 77.79° | (131) | 78.25° | (202) | 76.77° | (211) |
| 92.75° | (040) | 93.34° | (222) | 91.60° | (220) |

**IV: Piezoelectric Characterization of (002) AlN**

The work function of the electrode material may have an impact on the electrical properties of AlN.8 Our composites use standard (111) Pt as both the bottom and top electrodes, because previously (111) Pt was shown to promote a (002) AlN texture.9 The electrical properties (polarization and displacement) of Samples 1, 2, and 3 were obtained via double beam laser interferometer (DBLI) measurements. The samples were prepared into a parallel plate capacitor geometry by sputter depositing Pt through a 1000 μm diameter shadow mask. This resulted in a top electrode area ranging between 0.968 – 1.19 mm2. Previously, annealing AlN to temperatures as high as 500 °C was found to improve the piezoelectric performance. 10 We analyze the structural and piezoelectric properties of AlN in Samples 1-3 after annealing at 700 °C for 15 min. Polarization and displacement curves were taken at a minimum of three spots on each samples to generate an average *d*33,f at each applied voltage (i.e 10V, 20V, 50V, and 100V). The slope of the displacement curves at each applied voltage are used to obtain *d*33,f. The average of all values were used to obtain the mean *d*33,f ± standard deviation of each sample, as shown in Table SIII.

TABLE SIII: Average d33,f measurements obtained via DBLI on Samples 1 – 3 before (as deposited) and after annealing, varying the voltage between 10V – 100V

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Voltage | Sample 1: As-deposited | Sample 2: As-deposited | Sample 3: As-deposited | Sample 1 - Annealed | Sample 2 - Annealed | Sample 3 - Annealed |
| 10 V | 6.1 pm/V | 5.6 pm/V | 6.1 pm/V | 5.8 pm/V | 6.4 pm/V | 6.3 pm/V |
| 20 V | 5.9 pm/V | 6.0 pm/V | 6.3 pm/V | 5.7 pm/V | 6.2 pm/V | 6.5 pm/V |
| 50 V | 5.4 pm/V | 5.9 pm/V | 6.3 pm/V | 5.7 pm/V | 6.5 pm/V | 6.6 pm/V |
| 100 V | 5.6 pm/V | 6.3 pm/V | 6.5 pm/V | 6.4 pm/V | 6.4 pm/V | 6.2 pm/V |
| Average d33,f | 5.8 pm/V ± 0.3 pm/V | 6.0 pm/V ± 0.3 pm/V | 6.3 pm/V ± 0.2 pm/V | 5.9 pm/V ± 0.3 pm/V | 6.4 pm/V ± 0.1 pm/V | 6.4 pm/V ± 0.2 pm/V |

Figure S3 demonstrates the polarization and displacement curves taken at 100V (i.e electric field of ± 500 kV/cm) for Samples 1 – 3. Polarization is well-known to be dependent on grain size, domain structure, and domain mobility.8 The difference in the polarization hysteresis loop behavior between the as-deposited and after-annealed state of each sample is likely due to the variation in leakage current from changes in the grain size, surface roughness, and *c*-axis growth.8 Previously, it was found that grain boundaries act as pinning centers for polarization, thus polarization switching was easier for large AlN grains.8 In general, it was found a lower surface roughness of the bottom electrode results in a lower leakage current. All samples in the as-deposited state show similar d33,f,values and expected linear polarization behavior. In agreement with other studies,10 annealing AlN was found to improve the d33,f,values. However, upon annealing all samples, an open polarization loop is obtained which generally indicates a larger leakage current and higher resistive losses. The polarization behavior shown in Figure S3 is similar to the behavior of AlN grown on Ti, Pt, and Al electrodes.8 These results prompted investigation of the interfaces to correlate microstructure changes to the electrical and structural properties of AlN. Future studies should consider a more in-depth analysis of the underlying SMA effects on the electric properties of sputtered AlN films.

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**Figure S3:** Polarization and displacement curves obtained from DBLI measurements at 100V. Samples 1, 2, and 3 (a-c) in the as-deposited state and (d-f) after rapid thermal annealing (RTA).

**V: Micro-structural characterization of SMA ME composites**

**A. *In-Situ* Temperature Dependent TEM:**

The average structural transformation of the TiNiCu film was investigated by heating the samples *in-situ* to temperatures above the austenite finish temperature Af = 69 °C. The results are presented in Figure S4 showing the electron diffraction (ED) patterns and rotational average plots of the intensity distribution at room temperature and at 120°C, respectively. The phase transformation from the martensite into the parent austenite phase is evidenced for both films by smoothing of the ED patterns and the profiles. This is because the low symmetry orthorhombic B19 phase is transformed into the high symmetry cubic B2 austenite phase; thereby the number of reflections is significantly reduced. Prior studies on the phase transformation mechanism in Ti-rich TiNiCu films revealed Ti-rich nano-precipitates embedded in the base alloy to serve as epitaxial misfit strain mediators between the B19 and B2 phases.11 These precipitates promote the transformation upon temperature cycling by epitaxial stabilization of a B2 transition layer, even in the martensite stable regime. In addition, at least in the case for Sample 2 where the TiNiCu film crystallized prior to AlN deposition, the presence of larger precipitates presumably of the kind Ti2Cu and Ti2Ni was evidenced and contribute as non-active phases to the ED series. More details of the microstructure of Ti-rich TiNiCu films can be found in *Dankwort et al.*12 Hence, despite the presence of precipitate phases the *in-situ* analysis demonstrated the transformation of the active martensite to the austenite phase for both Samples 2 and 3.

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**Figure S4:** ED pattern of TiNiCu at room temperature and at 120°C and rotational average (RA) plots showing the structural change upon transformation. (a) Composite SMA was crystallized prior to AlN growth (Sample 2) (b) SMA was crystallized after AlN growth (Sample 3-annealed). After heating the films above the austenite finish temperature Af both samples show distinct smoothing of the RA plots indicating the structural transformation.

**B. *Discussion of Diffusion***

The formation of a diffusion region is sketched in Figure S5 resembling simplified experimental EDS concentration profile data. Herein, the stages of the envisioned diffusion process at the TiNiCu/Ta/Pt interface are depicted under following assumptions: The situation where Ta/Pt/ AlN is deposited after rapid crystallization of TiNiCu (Sample 2), via rapid thermal annealing (t < 20 s) is shown in Figure S5 (top). A Cu-poor and Ti-enriched interface region is formed immediately (blue box). Such interface morphology was also observed at the Si substrate/ Ta-adhesion interface layer in both investigated composites after 15 minutes of annealing. The situation where Ta/Pt/AlN is annealed with TiNiCu (Sample 3) promotes diffusion originated from the grains at the interface, resulting in a mixed polycrystalline region under complete dissolution of the electrode structure, shown in Figure S5 (bottom).

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**Figure S5:** Sketch showing the measured EDS concentration profile across the TiNiCu/Ta/Pt interface. (top) Sample 2, situation after rapid crystallization, no diffusion took place and a Cu-poor and Ti-enriched interface layer is established. (bottom) Sample 3-annealed, situation after diffusion, Ti diffuses towards the Pt layer and Ta, Pt diffuse towards the TiNiCu matrix. green box: TiNiCu matrix, blue box: Cu-poor and Ti-rich crystal interface, orange box: Ta-layer, red box: Pt-layer, violet box: diffusion region.

The complete interpretation of diffusion in a multicomponent system with only two sets of data is not possible. In the following, we will give a coarse idea of the diffusion mechanisms taking place along the following lines and boundary conditions.

Upon annealing, TiNiCu crystallizes immediately, as the 700 °C annealing temperature is much larger than the crystallization temperature of most NiTi films which is in the vicinity of 500°C.13 From TEM it is clear that all layers are polycrystalline with lateral grain sizes of around 200 nm in the TiNiCu alloy due to crystallization and around 10 nm and 100 nm in Ta and Pt layers (grain size on the order of the film thickness). This setup is regarded as an interdiffusion experiment in a multicomponent alloy with fine grain size.

To discuss this, thermodynamics (i.e. attractive and repulsive interactions of the up to five components, possible intermetallic phase formation, solubility limits) as well as kinetics (tracer and chemical diffusion in the bulk as well as grain boundary diffusion) must to be taken into account. We start by estimating the diffusivities needed to explain the change of the concentration profiles, using the well-known parabolic growth law14

with *t* = 15 min and = 100 nm being roughly half of the length of the diffusion zone (see Fig. S5) results in diffusivities of the order of m²/s.

However, it is not clear, whether bulk or grain boundary diffusion is dominant, with the latter being orders of magnitude faster than the former. No data for grain boundary diffusion in this system are available.15 Bulk diffusivities can be estimated from known data collections14 depending on matrix and diffusing element. Calculations result in diffusivities between m²/s for Fe in Ta and for Al in Ta (according to tables in *Neumann et al.*16*).* These values for bulk diffusivity are much too small compared to the needed m²/s. However, it must be stressed that grain boundary diffusion might occur as well and known to be three to five orders of magnitude larger than the bulk values calculated here.14,15,17 Thus, from kinetics and diffusion point of view, such concentration profiles as shown in Figure S5 are possible.

To understand the sequence of processes and the shape of the concentration profiles in detail, the thermodynamics of the binary systems serve as acceptable approximations to estimate solubility limits (thermodynamic factor of diffusion)14 in multiple phase systems. The binary phase diagrams describe for the system {Ti-Ta}: good solubility with two intermetallic phases (IPs),18 for {Ta-Pt}: little solubility and a large number of IPs,19 for {Ti-Pt}: little solubility with large number of IPs,20 for {Ni-Ta}: medium solubility and large number of IPs,21 and for {Cu-Ta}: no solubility.22

From the good solubility of Ti in Ta and the small grain size of the Ta-layer we expect that first the 10 nm Ta barrier is dissolved by interdiffusion via grain boundary diffusion. Then, without an intact Ta barrier, Ta and Pt start to diffuse towards the boundary of the Ti-enriched interface layer, Ta also diffuses into the Pt-layer. Formation of binary or ternary Ti-Ta-Pt phases having no solubility of Ni, causes a push-back of Ni towards the matrix resulting in a detectable Ni-enrichment there. Cu is also pushed back due to the low solubility of Ta in Cu. Note, the exact nature of phases formed within this diffusion region and the respective grain sizes have not been determined and are assumed to be strongly heterogeneous.

In summary, it is consistent with kinetics and phase diagrams that first crystallization occurs, then dissolution of the Ta barrier via grain boundary diffusion and then grain boundary diffusion of Ta and Pt into the remaining matrix pushing back Ni and Cu.

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