Superconductivity in $\text{Ti}_{67}\text{Zr}_{19}\text{Nb}_{11.5}\text{Sn}_{2.5}$ shape memory alloyM. Egilmez^{1,*}, O. Batal², W. Abuzaid², Z. Salman³, M. Alkhader², H. Akamine⁴,
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Shape memory alloys (SMAs) exhibit unique functionalities due to their superelastic and shape memory properties. The ability to program and alter their shapes following a thermomechanical stimulus makes them highly important materials for a vast number of applications in the aerospace, automotive, biomedical, and robotic sectors. Research on SMAs has largely focused on metallurgical, mechanical, structural, or phase transformation properties. Here, we investigate the electrical, magnetic, and thermodynamic properties of the biocompatible SMA, $\text{Ti}_{67}\text{Zr}_{19}\text{Nb}_{11.5}\text{Sn}_{2.5}$ (at. %). In particular, we report the discovery of a superconducting phase transition with a critical temperature of 4.65 K with 0 K critical magnetic fields of $H_{c1} = 13.7$ mT and $H_{c2} = 9.2$ T. From the temperature dependence of the specific heat and local magnetic field measurements using transverse field muon spin rotation, we also determine a superconducting coherence of 6 nm and a London penetration depth of 776 nm. The results are key towards the development of cryogenic electrical device applications of SMA materials.

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I. INTRODUCTION

Depending on their composition, thermal treatment, and deformation temperature, a shape memory alloy (SMA) exhibits superelasticity and/or a shape memory effect [1–3]. In their austenitic state, SMAs accommodate significant deformation through a reversible stress-induced martensitic transformation (i.e., superelasticity) [1,4]. Mechanical loading of the martensite phase results in the shape memory effect where retained strains are recovered following a thermomechanical or magnetic stimulus. A number of SMA materials exist including Ni-based alloys (e.g., NiTi and NiFeGa [5]), Cu-based alloys (e.g., CuZn and CuZnAl [6]), Fe-based alloys (e.g., FePt, FeMnNiAl [7]), AuZn [8] and Co-based alloys (e.g., CoNiAl [9]).

The near equiatomic SMA NiTi is one of the most widely studied compositions, in part because it can sustain superelastic strains up to 10% and shape memory functionality [10]. This SMA has wide-ranging applications from actuators in robotics [10,11] and morphing systems in aerospace applications [13] to biomedical stents [11–15]. However, a drawback of NiTi relates to the toxicity of Ni [16], which has motivated the development of alternative, biocompatible, SMAs, most notably TiNb-based materials [15,16]. The recently discovered ternary alloy TiNbZr has proven to be an excellent alternative to NiTi-based SMAs, exhibiting similar

mechanical properties [17–20], and improved biocompatibility. The superelasticity in TiNbZr-based materials can be finely tuned through thermomechanical treatment and via the addition of biocompatible elements such as Mo, Ta, and Sn [16,21–23], which stabilize the Austenite body centered cubic (bcc) structure. The Zr improves the superelastic response of TiNb-based SMAs at room temperatures [24] while alloying with Sn enhances the stability of the transformation stress when subjected to cyclic superelastic loading [25].

The majority of experimental studies on TiNb-based SMAs have focused on their metallurgical, mechanical, structural, and phase transformation properties. However, it is now accepted that studying their electrical transport properties is also very important which are key for the development of actuators [25–28] and functional devices [29]. Here, we report a detailed study of the low-temperature electrical transport properties of $\text{Ti}_{67}\text{Zr}_{19}\text{Nb}_{11.5}\text{Sn}_{2.5}$ (at. %) or “TZNS,” a biocompatible SMA [30] which can achieve recoverable strains up to 7%. The key result is that we observe a superconducting transition at 4.65 K with austenite to martensite phase transition between 150 and 225 K. Superconductivity has been observed in AuZn ($T_c \approx 1.21$ K [31]), InTi ($T_c \approx 2$ K [31]), and U-based ($T_c \approx 1.2$ – 2.0 K [32]) SMA systems. We note that superconductivity has been reported for a large compositional range for NbTi- and NbSn-based systems [31], but to our knowledge superconductivity has not been seen in TiNbZr-based SMAs. Superelastic behavior in TiZrNbSn shape memory alloy along with superconducting properties makes this material a promising candidate for

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FIG. 1. (a) X-ray diffraction trace of the TZNS alloy. (b) TEM bright-field image. (c),(d) Corresponding SAEDPs taken from [011] and [113] incident beam directions of bcc structure, respectively. (e)–(h) STEM-EDX maps of Ti, Nb, Zr, and Sn confirming the elemental homogeneity.

many superconductor applications such as high current carrying superconducting wires in the bulk form and electronic applications in the thin film form. It should be noted that the SMA properties of TiNbZr-based compositions require precise control of grain size to achieve room-temperature superelastic properties. This is generally achieved through plastic deformation of as-cast material to around 95% cold rolling [33,34]. The effect of such extreme deformation on the electrical transport properties including the superconducting transition temperature is the focus of this work

II. EXPERIMENTAL DETAILS AND STRUCTURAL CHARACTERIZATION

The TZNS ingot is arc melted from elements with purities better than 99.5 wt %. Following casting, the ingot is homogenized at 1100 °C for 24 h in a vacuum. Strips of TZNS are cut from the ingot and subjected to cold rolling to achieve a thickness reduction of 91–96.8%. A number of TZNS strips are then heat treated in vacuum encapsulated quartz tubes at temperatures between 300 °C and 950 °C for 30 min followed by quenching in water. A series of TZNS strips are investigated and compared: homogenized (A); as-rolled to different rolling levels (B); and heat-treated (C) (including homogenized, rolled, and heat-treated at different temperatures).

A Panalytical Xpert³ powder diffractometer is used for phase identification and lattice parameter determination. Microstructural investigations are carried out using a scanning electron microscope (Tescan Vega 3) and a scanning transmission electron microscope (STEM, JEM-ARM200F) operated at 200 kV and equipped with energy dispersive x-ray spectroscopy (EDX). The STEM specimen was prepared by focused ion beam (FIB, Thermo Fisher Scientific Versa 3D DualBeam) milling. Relaxation calorimetry is used for heat

capacity measurements. Electrical transport is performed on 0.07 mm × 0.35 mm × 2.5 mm rectangular TZNS strips using a pulse-tube closed-cycle physical property measurement system. Superconducting properties are also characterized utilizing transverse field muon spin rotation (μ SR) using the GPS spectrometer at the Paul Scherer Institute.

III. RESULTS AND DISCUSSION

In Fig. 1(a) we have plotted an x-ray diffraction trace from a solution-treated (900 °C for 30 min followed by a water quench) TZNS strip, which indicates single phase bcc or β phase with a lattice parameter of 0.338 (1) nm. The peaks at higher angles are broad, indicating a distorted lattice that is consistent with the different atomic radii of the constituent elements: the atomic radii mismatch between Zr and Ti is close to 9%. Figures 1(b) and 1(c) show the TEM bright field image and corresponding selected area electron diffraction pattern (SAEDP) taken from the [011] incident beam direction of the bcc structure, respectively. Figure 1(d) is the SAEDP taken from [113], which was tilted about 31° around the $[21\bar{1}]$ axis. All of the diffraction spots in Figs. 1(c) and 1(d) can be indexed as a bcc structure with a lattice parameter of 0.338(1) nm. There are no extra spots derived from secondary phase regions such as Nb clusters, ω and α precipitates. A high-resolution TEM image presented in the Supplemental Material [35] provides further evidence confirming the single bcc structure. Figures 1(e)–1(h) show STEM-EDX elemental maps corresponding to Fig. 1(b), revealing a uniform distribution of elements with a homogeneous chemical composition over the entire scan area. Several SEM-EDX scans are collected from different regions of the strips, at various magnifications, revealing the same compositional ratios as listed in Table I.

TABLE I. The nominal composition in wt% and at.% of the TZNS.

Ti (at. %)	Zr (at. %)	Nb (at. %)	Sn (at. %)
67.2	19.2	11.1	2.5
Ti (wt %)	Zr (wt %)	Nb (wt %)	Sn (wt %)
51.2	27.8	16.4	4.7

The electrical resistivity (ρ) of a representative TZNS strip (95% rolled, 900°C annealed for 30 min) versus temperature (T) is shown in Fig. 2(a). The rise in ρ with decreasing T shows a residual resistivity ratio of $\rho(300\text{ K})/\rho(5\text{ K})$ of 0.984, which is consistent with granular-metallic behavior [36–38], i.e., a metal-weak insulator composite below geometrical percolation threshold where metal grains with a bulk lattice structure are surrounded by an insulating grain boundary region that is thicker than the electron mean free path length for charge scatter. Hence, intergrain transport involves charge tunneling and hopping processes which lead to an increase in electrical resistance (R) at lower temperatures [38]. As indicated in the detailed microstructural analysis shown in Fig. 1, our specimens are polycrystalline samples that involve large amounts of grain boundaries. We have used the Scherrer equation to estimate the average crystallite size

from the broadening of the peaks (instrumental broadening is eliminated). Our analysis revealed that in our samples the average crystallite size varies between 27 (2) nm and 32 (2) nm, supporting the idea of large density of grain boundaries in our samples.

In general, the shape memory and superelastic behavior of SMAs are associated with a phase transition from martensite to austenite [39] (stress induced in the case of superelasticity and temperature induced for the shape memory effect). Such structural changes also affect the electronic properties [40] and a reversible change in resistance (R) of the SMA is seen across the T -phase transition [40]. Such a response of R to the structural phase transition can be attributed to so-called “superzone boundary gaps,” which affect the electronic density of states near the Fermi energy [35,38], and the martensite-to-austenite transition is visible in $R(T)$ in the 150–225 K range. Moreover, McDonald *et al.* linked the observation of resistance anomaly at the martensite to austenite transition to the rearrangement of the Fermi surface revealing the significance of the electronic band structure in SMA materials [29].

Figure 2(a) shows a sharp drop in $R(T)$ below 7 K with a transition to a superconducting state with an onset critical temperature of $T_{c0} = 4.65\text{ K}$ [Fig. 2(b)]. A transition is also observed in the T dependence of the specific heat in zero magnetic field [see inset of Fig. 2(b)]. In Fig. 2(c) we have plotted the T dependence of the zero field cooled (ZFC) and field cooled (FC) magnetization (M) of the TZNS strip in a

FIG. 2. (a) $\rho(T)$ in warming (w , left arrow) and cooling (c , right arrow). (b) $\rho(T)$ of a TZNS strip near the superconducting critical temperature. The inset shows the T dependence of the specific heat. (c) T dependence of the ZFC and FC magnetization measured in a magnetic field of 0.5 mT. (d) Representative strain-strain response showing superelasticity in the TZNS strip measured at 300 K. Arrows indicate forward and reverse transformation respectively.

FIG. 3. (a) $\rho(T)$ in different magnetic fields (labeled). (b) Upper critical field $H_{c2}(T)$ where the solid line is a fit to Eq. (1). (c) $M(H)$ at different T (labeled). (d) $H_{c1}(T)$ where the solid line is a fit to Eq. (2).

magnetic field of 0.5 mT. Consistent with transport and specific measurements, M measurements reveal a T_{c0} of 4.65 K.

Mechanical testing has been also performed on a matching cold rolled TZSN strip. Figure 2(d) shows a representative stress-strain curve of a TZSN strip deformed to about 2% strain in tension. A superelastic response is observed with a recovery of $\varepsilon_{\text{recoverable}} = 1.85\%$ and insignificant accumulation of residual strain of $\varepsilon_{\text{residual}} = 0.07\%$. Further details on the mechanical loading and the evolution of superelastic properties are shown in the Supplemental Material (see [35]). The behavior in Fig. 2 has been confirmed in 23 specimens of matching composition.

To investigate the superconducting state in the TZNS strip, we measured $\rho(T)$ in different magnetic fields as shown in Fig. 3(a), which shows a typical behavior of type 2 superconductivity. Values of T_{c0} versus magnetic field are determined from these measurements [Fig. 3(b)] from which we estimate the upper critical field of the TZNS from

$$H_{c2}(T) = H_{c2}(0)[1 - (T/T_c)^2], \quad (1)$$

to be $H_{c2}(0) = 9.20(4)$ T, which is comparable to the Pauli-paramagnetic limit for weak electron-phonon coupling of $H_{c2} = 1.86 T_{c0} \approx 8.65$ T [41–46]. From H_{c2} , we estimate a 0 K superconducting coherence length of $\xi(0) = 6(0.5)$ nm via $\xi(T) = \sqrt{\frac{\Phi_0}{2\pi H_{c2}(T)}}$, where Φ_0 is the flux quantum [47].

In Fig. 3(c) we have plotted $M(H)$ loops in the superconducting state in which H_{c1} is determined from the point of

deviation from linear $M(H)$ in the low H regime [48], showing behavior that closely matches

$$H_{c1}(T) = H_{c1}(0)[1 - (T/T_c)^2], \quad (2)$$

from which we extrapolate $H_{c1}(0) = 13.7(2)$ mT [Fig. 3(d)].

The T dependence of the specific heat measured in various magnetic fields is shown in Fig. 4(a). In ZF, a discontinuity of $\Delta C = 49.05$ mJ/mol K is observed at $T_{c0} = 4.65$ K. It is well known that the lattice specific heat does not change at the superconducting transition temperature. Hence, the jump in specific heat at T_c is due to changes in the electronic specific heat. At 9 T, the T_{c0} was reduced beyond the measurement of capability of our experimental setup. Therefore, the T dependence of the specific heat at 9 T can be regarded as the normal state specific heat. Assuming that the normal-state low- T specific heat is given by $C = \gamma T + \alpha T^3$, where γ and α are the electronic and lattice specific heat coefficients [49], we obtain the coefficients γ and α using a linear fit when low temperature specific heat is written in the $C/T = \gamma + \alpha T^2$ form. Figure 4(b) shows the specific heat values in zero and 9 T fields (C/T versus T^2 plot). A linear fit to the 9 T specific heat data in Fig. 4(b) gives $\gamma = 7.36$ mJ/mol K² and $\alpha = 0.51$ mJ/mol K⁴. In contrast to the 9 T data, in zero field, data reveal bulk superconductivity where the entire specimen superconducts below T_{c0} . The transition from the normal state to the superconducting state can be seen as a disorder-order transition and the entropy loss in this process is $\Delta C/\gamma T_c = 1.43$ if the superconductivity is a bulk effect

FIG. 4. (a) T dependence of specific heat in selected magnetic fields (labeled). (b) Normalized specific heat at zero field and 9 T as a function of T^2 .

[49]. Note that this value was revealed by the Bardeen Cooper Schrieffer (BCS) theory for weak coupling superconducting materials, and is found to be the case for the majority of superconductors [49]. Given that $\Delta C = 49.05 \text{ mJ/mol K}$ and $\gamma = 7.36 \text{ mJ/mol K}^2$ one can obtain $\Delta C/\gamma T_{c0} = 1.4315$ for the considered SMA in this work. This value is in line with BCS theory predictions. We also estimate a Debye temperature of 156.2 K using $\theta_D = (12\pi^4 R/5\alpha)^{1/3}$, where R is the ideal gas constant [49].

Superconductivity is further confirmed by transverse field μSR measurements. In these measurements, fully spin polarized positive muons (μ^+) are implanted into the studied sample, with a magnetic field applied transverse to the initial spin direction. Muons decay into a positron with a lifetime of $\tau_\mu \sim 2.2 \mu\text{s}$, which is emitted preferentially in the direction of the muon's spin at the time of decay. The ensemble average of the polarization of the implanted muons is proportional to the measured time dependence of asymmetry $A(t)$, which is monitored via the asymmetric decay using appropriately positioned detectors. $A(t)$ is used to extract internal magnetic field information at the muon site.

To measure the superconducting properties of the material, the sample is cooled in an applied field of 50 mT, while measuring $A(t)$ as a function of temperature. $A(t)$ measured at 1.5 and 10 K are shown in Figs. 5(a) and 5(b), as representative of such data. Below T_{c0} , a vortex lattice is formed in the superconducting sample which results in an inhomogeneous broadening of the field distribution sensed by the muons. To quantify this broadening versus T , we fit $A(t)$ to a Gaussian damped oscillation using the MUSRFIT package [50]. We have used the Gaussian function as $G_z(t) = A \cos(\gamma_\mu B t + \phi) \exp(-\frac{\sigma^2 t^2}{2})$, where A is the initial muon asymmetry, $\gamma_\mu = 2\pi \times 135.5 \text{ MHz/T}$ is the gyromagnetic ratio of the muon, B is the mean local magnetic field sensed by the muons, σ is the damping rate of the precession amplitude which reflects the width of local field distribution, and ϕ is the initial phase which depends on the initial spin orientation and detector. Solid lines in Figs. 5(a) and 5(b) are fits to the Gaussian function. The temperature dependence of σ and B are shown in Fig. 5(c) and the inset respectively. The inset of Fig. 5(c) shows the mean field obtained from the fits: a clear diamagnetic response is visible. B exhibits a clear diamagnetic response below T_{c0} which coincides with

FIG. 5. (a),(b) Time dependence of the asymmetry $A(t)$ at 1.5 K (a) and 10 K (b). (c) T dependence of the damping rate. The solid line is a fit to BCS theory's predicted energy gap equation. Inset: Mean-field distribution obtained from the fits.

FIG. 6. (a)–(c) Temperature dependence of the resistance at indicated cold work fractions. (d) T_c dependence of the heat treated specimens with 95% cold work fraction. Here P represents the pristine (undeformed sample) and AR represents as-rolled sample. Arrows indicate the direction of the temperature sweep.

the increase in σ . In the limit of a small applied field, σ is related to the London penetration depth in a superconductor, such that $\sigma_{SC}(T) = \sqrt{\sigma^2 - \sigma_N^2} = 0.0609\gamma_\mu\phi_0/\lambda^2$ where ϕ_0 is the magnetic flux quantum, σ_N is the broadening in the normal state determined from measurements above T_c , and λ is the London penetration depth under the assumption that the whole sample is superconductive [51]. Note that the T variation of the superfluid density is modeled using

$$\frac{\sigma_{sc}(T)}{\sigma_{sc}(0)} = 1 + \frac{1}{\pi} \int_0^{2\pi} \int_{\Delta(\varphi, T)}^{\infty} \frac{\partial f}{\partial E} \times \frac{EdE d\varphi}{\sqrt{E^2 - \Delta^2(\varphi, T)}}, \quad (3)$$

where f is the Fermi function, E is the quasiparticle energy, and $\Delta(\varphi, T) \simeq \Delta(\varphi) \tanh c_0 \sqrt{a_G(\frac{T_c}{T} - 1)}$ is the temperature dependence of the energy gap function in which the constants c_0 and a_G depend on the pairing state. For an s wave (BCS) superconductor constants can be taken as $c_0 = 1.785$, $a_G = 1$, and $\Delta(\varphi) = \Delta_0$. The fits to the BCS model using Eq. (3) are shown in Fig. 5(c) as solid lines, which yield an isotropic energy gap estimation of 0.98(9) meV. Details of similar calculations can be found in Refs. [52,53]. On the other hand, the BCS theory prediction $\Delta_0 = 1.764k_B T_{c0}$ to a value of 0.70(2) meV where k_B is the Boltzmann constant. Note that in the established fits to the BCS model, the T_{c0}

of the sample was taken as 4.3 K. It is not unusual that T_{c0} measured using μ Sr measurement is slightly lower than the T_{c0} determined from the bulk characterization measurements. Based on the measured σ we have obtained $\lambda_0 = 776(2)$ nm for the zero-temperature penetration depth through our fits. Using such a value one can estimate the Ginzburg Landau parameter of our shape memory alloy as $\kappa = \lambda/\xi = 129$. The calculated κ is higher than the NbTi and NbSn₃ alloy κ values [49].

Well documented superelastic behavior in TZNS along with the superconducting properties reported here demonstrates that this material is a promising candidate for bulk superconductor applications. The critical current density of TZNS at 4.2 K (1.3×10^6 A/m²; see the Supplemental Material [35]) revealed promising results for such applications. For high current carrying bulk applications, the T_{c0} of the TZNS seems to be low when compared to the widely used NbTi alloy. However, in our view, higher superconducting transition temperatures with desired SMA properties can be obtained through a detailed compositional study for this family of materials. Moreover, shape memory alloys are already being used in many sensor and actuator applications. Adding superconducting functionality to superior mechanical properties of SMA may lead to enhanced functional devices. 23 specimens were investigated in this work with different cold

rolling levels and annealing treatments. Such treatments are targeted to optimize the mechanical and superelastic properties of the SMA; however, the effect on the superconducting transition was also evaluated. As shown in Fig. 6 (selected cases), independent of the degree of rolling and subsequent thermal annealing, the superconductivity remained robust T_{c0} averaging around 4.41 to 5.09 K. We have measured the temperature dependence of the resistivity in both warming up (up arrows) cycles and cooling down (down arrows) cycles [Figs. 6(a)–6(c)]. In these measurements, the degree of rolling is given in percentages where the number (for instance 95%) indicates the percentage of change in the thickness of the rolled specimen. Here different levels of cold work will lead to a different level of disorder in the sample, in addition to affecting the resulting grain size. Note that the effect of the disorder on the superconducting properties is an important probe of the superconducting order parameter. Anderson theorem [54] postulates that disorder can depair electrons only when the superconducting gap is anisotropic or disorder is strong enough to break the time-reversal symmetry. Our magnetic, specific heat, and muon spin rotation analysis suggest that TZNS is a typical s -wave superconductor. In this respect the TZNS system (being a typical isotropic s -wave BCS system) is expected to be robust against disorder. In general, the cold work will result in extended defects and residual stresses within the sample which can be partially or fully lifted through thermal annealing. A representative example of thermal annealing on 95% rolled TZNS is shown in Fig. 6(d). As seen in the figure there is a slight variation in the T_{c0} (4.41 to 5.09 K) compared to the $T_{c0} = 4.65$ K of the as-received sample. Such T_{c0} measurements following heat treatment support the robust superconductivity in TZNS. The residual strain and defect density dependence of the superconducting transition temperature requires extensive microstructural and electrical transport measurements and analysis and is not within the scope of the current study.

IV. SUMMARY AND CONCLUSIONS

In this work, the electrical transport properties of the TZNS SMA are investigated. Temperature dependence of the resistance of the alloy exhibits a negative coefficient of resistance between room temperature and 7 K and a superconducting transition around 4.65 K. Temperature dependence of the resistance hinted at the austenite to martensite phase transition in the range 150–225 K. A detailed analysis of the electrical and magnetic properties reveals 0-K critical magnetic fields of $H_{c1}(0) = 13.7(2)$ mT and $H_{c2}(0) = 9.20(4)$ T. Detailed specific heat and transverse field muon spin rotation analysis revealed bulk superconductivity with an estimated superconducting coherence of $\xi = 6(0.5)$ nm and London penetration depth of $\lambda_0 = 776(2)$ nm. The reported superconducting states for the considered SMA were consistent at different levels of cold rolling and thermal treatments. We believe well-documented superelastic and shape memory behavior in TZNS SMA along with the superconducting properties reported here makes this material a promising candidate for many superconductor applications such as high current carrying superconducting wires. The hallmark of SMA functionality, namely the austenite to martensite transition, can be tuned to lower and higher temperature ranges through a detailed compositional study and cold work mediated microstructural modifications for this family of materials. Such tunability may open up new avenues for superconducting SMA devices.

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