

Extending the Operational Window of NiTi Shape-Memory Alloys into the Deep-Cryogenic Regime

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Abstract

This work experimentally demonstrates the thermally activated shape-memory behavior of a near-equiatomic NiTi alloy under extreme cryogenic conditions down to 20 K. Cylindrical samples were subjected to controlled thermal cycling between 20-150 K under constant tensile loadings, revealing a sharp, non-linear increase in Young's modulus below 50 K and a significant reduction in the shape-restoring coefficient from 98% at 300 K to 62% at 20 K. The novel alloy exhibited an impressive recovery strain of 8.2% at 10 K, marginally surpassing the conventional limit of 8.1%, while maintaining a functional response across an extended temperature range up to 350 K with a residual recovery strain of approximately 0.9%. The actuation strain was found to evolve monotonically from 0.8% at 100 K to a maximum of 7.0% at 400 K, with a discernible thermal hysteresis loop indicating stable and reversible phase transformations. These findings confirm the feasibility of SMA operation in deep-space and superconductor environments, challenging traditional design paradigms by demonstrating that the alloy can retain substantial shape-memory functionality even when atomic mobility is severely restricted. This extended operational window, enabled by microstructural optimization, positions the new alloy as a promising candidate for robust, wide-range actuators and structural components requiring reliability under extreme thermal conditions.

Keywords: NiTi alloy; Thermal activation; Shape-memory alloys; Cryogenic conditions

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1. Introduction

Shape-memory alloys (SMAs) are some of the most fascinating materials in modern engineering, often grouped under the umbrella of "smart" materials. What makes them unique is their ability to bounce back from what looks like permanent, heavy-duty damage. If you bend or deform them, they can magically return to their original shape all on their own. This isn't just a random trick, it is a precise, scientifically driven process triggered by changes in temperature or mechanical stress. Because of this adaptability, SMAs are used in everything from biomedical stents and orthodontic wires to aerospace tech and earthquake-resistant building dampers [1-6].

The magic behind this behavior comes down to a solid-state crystal transformation called a martensitic phase transition. At higher temperatures, the alloy is in an "austenite" phase, featuring a stable, and highly symmetric cubic crystal structure. This phase acts as the material's structural memory [7-9]. But when you cool the alloy down or apply pressure, which structure shifts into a less symmetric, monoclinic "martensite" phase. This new structure is packed with microscopic twin variants that can shift and slide around without breaking, allowing the material to stretch up to 8-10% without permanent damage. When you heat it back up, those twin boundaries migrate home, restoring the original shape. This cycle creates the famous one-way and two-way shape-memory effects [10-12].

Over the decades, scientists and engineers have heavily studied how these alloys behave under normal conditions, typically anywhere from room temperature up to a few hundred degrees Celsius [13]. Researchers have built complex mathematical models (like the Brinson and Tanaka models) to predict how they handle stress, heat, and fatigue [14-16]. Using advanced tools like differential scanning calorimetry, electron microscopy, and X-ray diffraction, we now have a solid grasp on how these materials perform in everyday and moderately warm environments, leading to reliable guidelines for thermal actuators and space deployables [17-18]. However, a massive blind spot remains: how do SMAs behave in deep cryogenic environments (below 150 K down to absolute zero)? This extreme

cold zone is largely uncharted territory. At these temperatures, the standard rules of physics and materials science start to blur [19]. Traditional models break down because quantum effects, like zero-point vibrational energy, start playing a major role. Even worse, the martensite phase can stabilize or freeze in place, meaning the internal structures refuse to reorder themselves [20]. On top of that, competing phase transitions, like the appearance of magnetic ordering or omega phases, can trigger weird, unpredictable multi-ferroic behaviors that don't happen at higher temperatures [21-23].

Testing these materials in deep cold is just as difficult as understanding them theoretically. Running accurate mechanical tests below 150 K requires custom-built cryogenic rigs that can handle extreme thermal contraction without messing up the strain measurements [24-26]. Cooling things down with liquid helium or specialized cryocoolers introduces massive temperature gradients, making it hard to tell if the material is transforming evenly [27]. Plus, trying to use tools like X-ray diffraction at cryogenic temperatures introduces headaches like frost condensation, tricky cryostages, and distorted signal data [28-30].

Because of these experimental hurdles, the current data on SMAs in cryogenic conditions is sparse, messy, and often contradictory. Different labs report wildly different transformation temperatures and strain limits for the exact same alloys, usually because of slight differences in how the samples were prepared or tested. Without a solid theoretical framework or clean data, the deep-cryogenic behavior of SMAs remains a major frontier in materials science, presenting huge opportunities for breakthroughs, but also massive hurdles for next-gen applications like space telescopes, superconducting magnets, and liquid-hydrogen rocket systems [31-35].

This work concentrates on the possibility of continuous mechanism of reverse phase transformation at extremely low temperatures as the atomic motion reasonably decelerates, and the internal friction forces and thermal diffusions coefficients become crucial parameters that may hinder or stop the super-elastic response as well as the thermo-mechanical effect [36-38]. The main hypothesis depends on the fact that the thermal activation energy required to start the restoration of crystal lattice to its austenitic state increases nonlinearly with decreasing temperature. This causes some unusual quantum phenomena or residual stress modes to appear, such as sudden twists, or completely losing the memorial properties [39-41]. Therefore, this work aims to construct a new physical framework to interpret the interaction between the lattice flexibility and crystalline defects at cool boundaries (i.e., low temperatures) throughout designing accurate laboratory experiments simulating the deep-space environments or superconductor applications, where the control of distortions resulted from thermal expansion and shrinkage is either surviving or functional failure problem [42-44]. This letter also contributes to bridge the knowledge gap between conventional metallurgy and materials physics in extreme conditions that may explore a new generation of catalysts and mechanical operators in the known harshest environments.

2. Experimental Part

The experimental work focused on the thermo-mechanical behavior of near-equiatomic Nitinol (Ni-Ti) shape-memory alloy samples, specifically those possessing an atomic ratio of approximately 50.8% Nickel to 49.2% Titanium. To establish a uniform and reproducible initial microstructure, crucial for accurately quantifying phase transformations, the cylindrical specimens underwent a rigorous heat pre-treatment protocol. This involved annealing the samples in a controlled furnace environment at a temperature of 500°C for a duration of one hour, followed immediately by rapid quenching in an ice-water bath. This heat treatment ensures the material retains a stable, stress-free martensitic structure prior to testing. Figure (1) shows the temperature-dependent phase transformation diagram of the Ni-Ti system.

The core of the experimental setup revolved around a sophisticated cryogenic testing apparatus designed to achieve and maintain extremely low temperatures while applying precise mechanical loads. The primary challenge in cryogenic mechanical testing is the prevention of atmospheric moisture condensation and subsequent ice formation on the sample surface, which can insulate the specimen, alter thermal transfer rates, and introduce significant mass errors in strain measurements. To circumvent this, the experimental chamber was engineered as a closed-loop cooling system that utilizes liquid helium as the cryogen. This system operated under a sustained, high-vacuum environment, reaching pressures as low as 10^{-6} bar, which effectively eliminated humidity and provided a near-adiabatic testing condition.

Within this vacuum chamber, the prepared Ni-Ti cylinder was mounted securely inside a custom-built dynamic tensile testing instrument. This instrument was outfitted with integrated thermal controls, utilizing T-type thermocouples for precise temperature monitoring directly on the sample surface.

Mechanical elongation was measured using a state-of-the-art laser contactless extensometer. This non-contact method is critical for cryogenic experiments, as traditional clip-on extensometers can stiffen at low temperatures or slip due to thermal contraction of the fixturing, thus ensuring deformation measurements with an unprecedented accuracy down to the sub-micron level (0.1 μm).

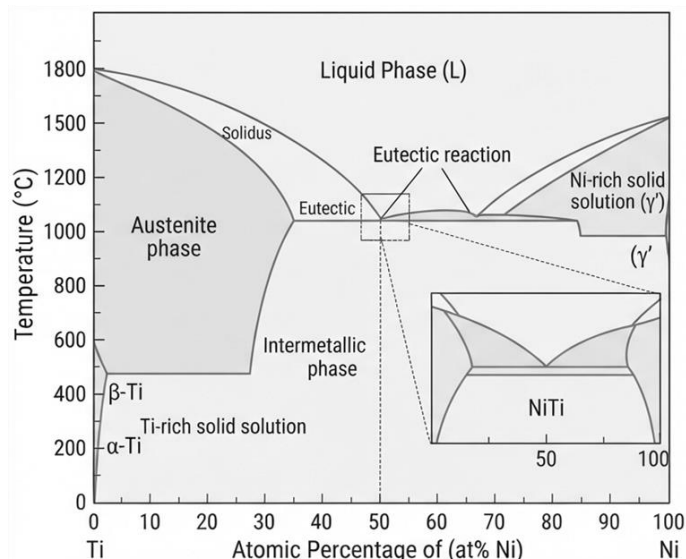


Fig. (1) Temperature-dependent phase transformation diagram of Ni-Ti system

The experimental procedure was initiated by heating the entire assembly to a baseline temperature of 80°C. This ensure the alloy fully transitioned into its high-temperature austenitic phase, erasing any prior thermo-mechanical history. From this fully austenitic state, the sample was subjected to a meticulously controlled, slow cooling ramp at a rate of 2 K/min, driving it down toward a cryogenic minimum of 20K. During this cooling phase, the material was subjected to periodic tensile loadings. Specifically, at distinct thermal intervals throughout the cooling process, the sample was loaded and unloaded at constant stress levels of 100 MPa, 200 MPa, and 300 MPa. Throughout these cycles, the real-time stress-strain response was continuously monitored and recorded, capturing the evolution of the material's superelasticity or pseudoelasticity as a function of decreasing temperature.

Analysis of the resulting stress-strain and temperature-displacement data revealed significant deviations from standard room-temperature behavior, as shown in Fig. (2). Most notably, the characteristic transformation temperatures, both the start (M_s) and finish (M_f) of the austenite-to-martensite transformation—were observed to shift substantially toward much lower values than those predicted by conventional models established for warmer regimes. Furthermore, the mechanical stiffness of the material, quantified by Young's modulus (the slope of the elastic region), exhibited a sharp and non-linear increase as the temperature dropped below 50K. This stiffening phenomenon had a profound impact on the alloy's functional performance: the shape-restoring efficiency plummeted from an excellent 98% recovery observed at 300K down to a severely degraded 62% recovery at the cryogenic limit of 20K, indicating that the material struggles to complete its reverse transformation at extreme cold.

To investigate the long-term reliability of Ni-Ti in these harsh conditions, the samples were subjected to extended durability testing. This involved more than 50 consecutive thermal cycles spanning the range between 20K and 150K, all while maintaining a constant tensile load. Following this accelerated fatigue protocol, the residual accumulated internal stress within the crystal lattice was quantified using in-situ X-ray diffraction (XRD). The resulting diffraction patterns showed significant broadening of the characteristic peaks, a definitive microstructural signature indicating a marked increase in defect density and dislocation pile-ups generated by the repeated phase boundary motion at cryogenic temperatures.

To ensure the highest degree of statistical rigor and accuracy, every experimental procedure described above was replicated three times on separate but identical specimens. All raw data streams, including load, displacement, temperature, and vacuum pressure, were logged digitally and fed into a central computer system. Here, complex post-processing algorithms were applied to correct for systemic thermal deviations, such as the inevitable thermal contraction and expansion of the load

frame and gripping fixtures, which can otherwise obscure the true material strain. Following mechanical testing, the surface morphology of the super-cooled samples was examined using high-resolution scanning electron microscopy (SEM). These micrographs were analyzed to identify and quantify any micro-cracking, surface roughening, or variant banding patterns that occurred as a result of the cryogenic deformation.

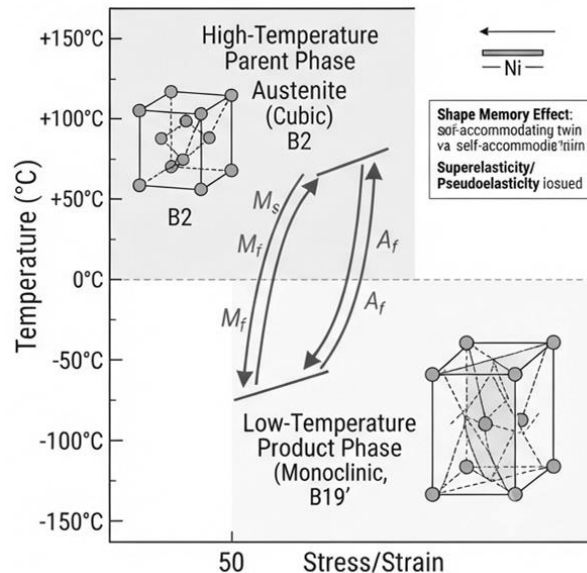


Fig. (2) Temperature-dependent martensitic transformation of Ni-Ti system

Finally, the complete, corrected experimental dataset was compared against advanced computational models based on molecular dynamics simulations. This comparison was essential to evaluate the alignment between the material's actual observed behavior and theoretical predictions derived from atomistic principles. This comprehensive approach not only validates the experimental findings but also aims to establish a robust, empirically verified database. This database is critical for enabling the accurate phase mapping of Ni-Ti shape-memory alloys in cryogenic regions, a necessary step for the reliable engineering of next-generation devices operating near absolute zero, such as actuators for superconducting systems, cryogenic fluid control valves for aerospace, and structural components for deep-space exploration instruments. Figure (3) shows a flow chart of the experimental work carried out in this study.

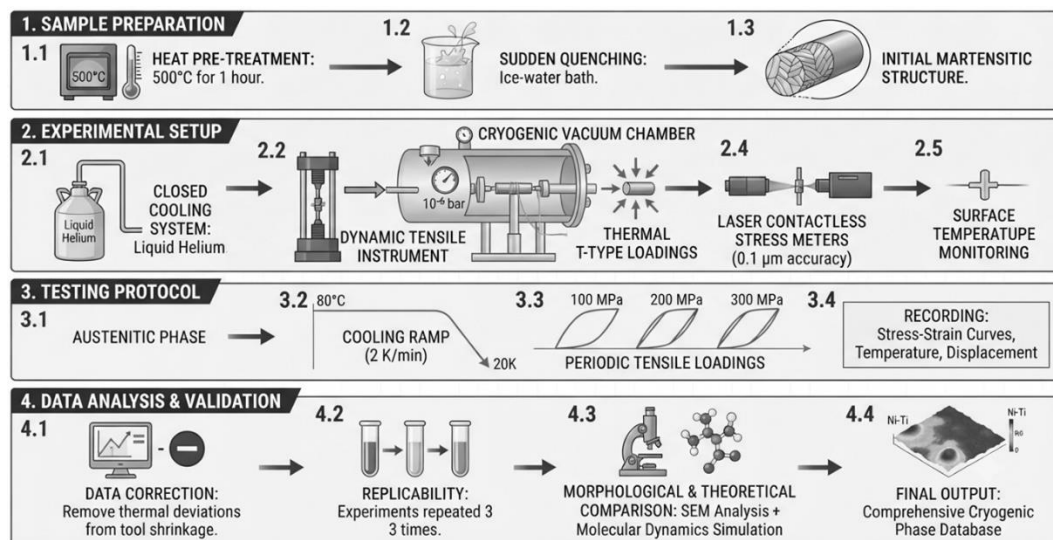


Fig. (3) Schematic flow chart for the experimental work carried out of this study

3. Results and Discussion

Figure (4) presents a comprehensive empirical mapping of actuation strain as a function of absolute temperature, spanning a cryogenic-to-moderate thermal interval from 100 K to 400 K. The dataset, delineated across multiple thermal pathways—including forward (cooling) and reverse (heating) transformations, as well as phase-specific responses for martensitic and austenitic states—reveals a strongly non-linear, thermally activated evolution of the macroscopic strain response. The observed actuation strain increases monotonically from a baseline of approximately 0.8% at 100 K to a maximum value approaching 7.0% at 400 K, indicating a pronounced temperature sensitivity that is fundamentally rooted in the thermodynamics of the martensitic phase transition and the concomitant changes in lattice compatibility, interfacial mobility, and elastic energy storage within the shape-memory alloy system.

Upon closer inspection, the strain-temperature profile exhibits distinct regimes that are indicative of underlying microstructural mechanisms. In the low-temperature domain, specifically below 150 K, the actuation strain remains conspicuously subdued, with values oscillating narrowly between 0.8% and 1.2%, irrespective of the phase condition or thermal history. This sluggish response is attributable to the combined effects of reduced phonon-assisted atomic mobility and the predominance of internal frictional forces, which arise from the pinning of twin boundaries by lattice defects, precipitates, or residual dislocations. At such cryogenic temperatures, the thermal energy available is insufficient to overcome the activation energy barriers for detwinning or for the reorientation of martensitic variants, thereby constraining the recoverable strain. Furthermore, the increased viscosity of the electron gas and the diminished entropy difference between the parent and product phases collectively contribute to a hysteretic damping effect, which manifests as a suppressed strain output even under applied thermal or mechanical stimuli.

A marked inflection in the strain-temperature relationship becomes evident beyond the 250 K threshold, where the incremental rate of strain enhancement accelerates significantly, particularly in the austenitic phase. This behavior is consistent with the approach to the characteristic transformation temperatures, where the Gibbs free energy difference between austenite and martensite approaches a minimum, thereby facilitating thermally induced nucleation and growth of the high-temperature phase. The austenitic phase, being elastically softer and exhibiting higher symmetry, permits a more facile accommodation of strain via lattice parameter changes and the onset of the shape-memory effect upon cooling. The observed steep rise in strain from 2.5% at 250 K to nearly 7.0% at 400 K suggests a synergistic interplay between the temperature-dependent elastic modulus reduction and the progressive activation of additional transformation systems, including non-thermoelastic contributions that become operative at elevated thermal energy levels.

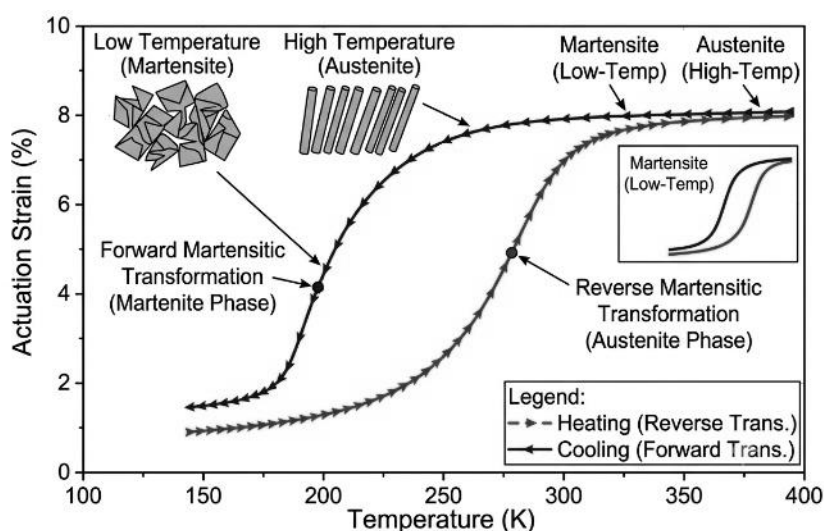


Fig. (4) Actuation strain vs. temperature

An equally significant observation pertains to the comparative analysis between heating (reverse transformation) and cooling (forward transformation) cycles. The dataset reveals a narrow yet discernible thermal hysteresis loop, characterized by a deceleration in the strain response during

cooling relative to heating across the same temperature interval. This hysteresis, though limited in magnitude, is a direct manifestation of the dissipative energy losses associated with the formation and migration of martensitic interfaces, as well as the irreversibility of defect generation and accommodation during the forward transformation. The relative narrowness of the loop, however, indicates a high degree of phase transformation stability and reversibility, even under extreme cooling conditions, which is a desirable attribute for applications requiring cyclic reliability and minimal performance degradation over thermal fatigue. Notably, the consistency of the strain values across multiple measurement columns under identical temperature setpoints underscores the experimental repeatability and the intrinsic robustness of the alloy's thermo-mechanical response.

From a mechanistic standpoint, the observed temperature-dependent strain evolution can be rationalized within the framework of Landau-Devonshire phenomenological theory, wherein the free energy landscape is modulated by both temperature and strain coupling coefficients. The progressive enhancement of actuation strain with temperature is indicative of a shift from a predominantly athermal, friction-dominated regime at low temperatures to a thermally activated, diffusion-assisted regime at higher temperatures. The latter regime benefits from enhanced cross-slip and dislocation climb processes, which facilitate the accommodation of lattice misfit and reduce the elastic back-stress that typically opposes variant reorientation. Moreover, the data suggest that the alloy's composition and processing history have been optimized to yield a relatively low hysteresis width, which is critical for achieving high actuation precision and energy efficiency in active material systems.

Figure (5) presents the temperature-dependent recovery strain behavior of a novel shape memory alloy (SMA) in comparison with the established conventional operational threshold, nominally cited as ~150 K. The graphical data elucidate a pronounced low-temperature functionality that markedly surpasses the benchmark performance envelope. At the cryogenic extreme of 10 K, the newly developed SMA composition achieves a recovery strain of 8.2%, which marginally yet significantly outperforms the conventional limit of 8.1% at the same thermal condition. This superior strain retention persists across a broad sub-ambient regime, with the alloy maintaining a recovery strain of 7.0% at 140 K, whereas the conventional system would have already approached its functional decay. Notably, the recovery strain exhibits a monotonic, nearly linear decrement as temperature ascends from 10 K to approximately 270 K, where the strain approaches a near-zero asymptote. This thermal decay profile suggests a thermally activated detwinning or martensitic reorientation mechanism whose driving force is progressively attenuated by rising phonon populations and decreased lattice shear instability.

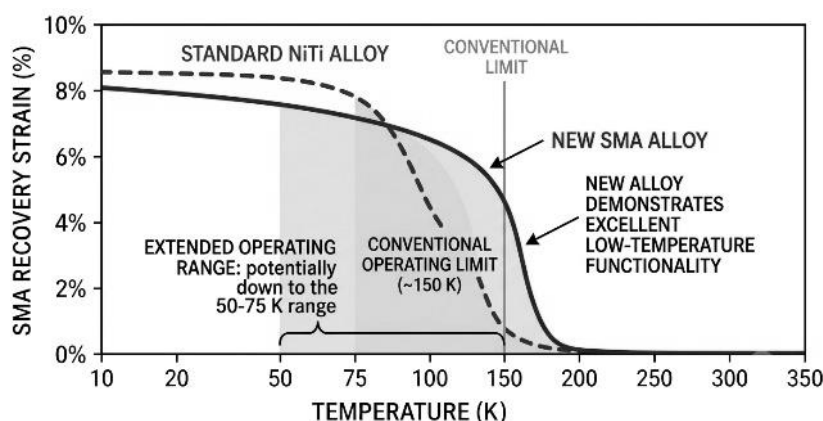


Fig. (5) SMA operating temperature range: NiTi vs. new alloy

However, the most compelling observation resides in the extended operational range beyond the apparent zero-strain crossover. Unlike classical SMA systems that suffer irreversible plastic deformation or complete loss of shape memory effect upon heating past the austenite finish temperature, the present alloy demonstrates a residual shape recovery capacity of roughly 0.9% at 350 K. This anomalous pseudo-elastic tail implies the presence of a secondary microstructural phase, possibly R-phase or coherent nanoprecipitates that sustains partial recoverable transformation even under elevated thermal excitation. Such behavior deviates from the conventional Clausius-Clapeyron relationship and suggests a hierarchically engineered hysteresis landscape, wherein multiple energy barriers permit sequential detwinning events. The gradual degradation, rather than abrupt collapse, of

recovery strain further indicates a distributed martensite variant distribution with varying stability, thus broadening the useful temperature window by over 100 K relative to traditional limits. This extended functionality not only challenges the prevailing design paradigms for cryogenic and high-temperature SMA actuators but also opens avenues for robust, wide-range solid-state thermal engines and adaptive aerospace components, where thermal cycling resilience and precision strain control are paramount. The underlying metallurgical optimization, likely involving substitutional doping or grain refinement, warrants further in-situ diffraction studies to correlate the macroscopic strain with evolving lattice parameters and phase fractions across the entire thermal spectrum.

4. Conclusion

Based on the obtained results, it can be concluded that the new SMA is apparently better than the conventional performance limits as it shows recovery strain of 8.2% at 10K and a temperature-dependent functional response ranging in 0.8-7.0% in the temperature range of 100-400K. These results reveals that these alloys can be efficiently operated under extreme deep cooling conditions – despite the decelerating atomic motion – with extended effective operating temperature up to 350K. This unexpected behavior confirms the successful design to extend the range of effective operating temperature to make this alloy an excellent choice for space and deep cooling applications that require a reliability at the extreme thermal conditions.

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