Physical and Mechanical Properties of LoVAR: a new lightweight particle-reinforced Fe-36Ni alloy

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ABSTRACT

Fe-36Ni is an alloy of choice for low thermal expansion coefficient (CTE) for optical, instrument and electrical applications in particular where dimensional stability is critical. This paper outlines the development of a particle-reinforced Fe-36Ni alloy that offers reduced density and lower CTE compared to the matrix alloy. A summary of processing capability will be given relating the composition and microstructure to mechanical and physical properties.

Keywords: LoVAR, Metal Matrix Composite, Fe-36Ni alloy, Invar®, dimensional stability, CTE

1. INTRODUCTION

Due to its low thermal expansion, Fe-36Ni finds extensive use in space structures that require high pointing accuracy and dimensional stability in the presence of dynamic thermal disturbances. The Fe-36Ni alloy and more recently the Fe-32Ni-5Co Super-Invar alloy provide the stability needed to help capture the images and spectra we have come to expect from observatories such as the Hubble Space Telescope, Kepler, and the upcoming James Webb Space Telescope. Low thermal expansion alloys such as Fe-36Ni are vital for the scientific payloads of satellites; however, they suffer from several drawbacks: low strength, low specific stiffness and subsequent mass penalties that result. Reducing the density (and by extension the overall mass of launch structures) as well as improving the thermal expansion and specific properties of these alloys would significantly improve the image quality and spectral resolution from future observatories allowing for greater scientific accuracy. The work presented in this paper outlines the requirements and results from the addition of a low coefficient of thermal expansion ceramic into the Fe-36Ni matrix. The formation of this ex-situ metal matrix composite (MMC) with improved physical and mechanical properties introduces a new class of dimensionally stable materials.

2. BACKGROUND

Increasing the performance of spacecraft structures is always the aerospace engineer's objective. Within the context of this work, the goals set for the metallurgist are familiar: decrease weight, maintain ductility, and improve dimensional stability. The guiding paradigm: Processing -> Structure -> Properties -> Performance provides the framework through which these goals are achieved. Achieving this enhancement requires a tailoring of the upstream processing to achieve a lighter, damage tolerant, and stable microstructure.

The metal matrix alloy for this initial effort is chosen from the binary Fe-Ni with Ni composition at 36 weight percent to minimize thermal expansion. Henceforth this will be referred to as Fe-36Ni. The low CTE and chemically stable Si_3N_4 was chosen as the ceramic. Incorporating a uniform dispersion of ceramic into the Fe-36Ni metal matrix is a requirement for ductile tensile properties. Achieving a high volume fraction of Si_3N_4 without introducing contamination that degrades dimensional stability presents a challenge. Simple twin-cone blending of powders causes triboelectric charging of the Si_3N_4 causing the powder to clump. This produces large agglomerates, as shown in Figure 1. Some approach ~1 mm in diameter.

A vastly improved approach employing mechanical alloying (MA) was used at Materion Aerospace Metal Composites' powder metallurgy facility in Farnborough, UK. Mechanical alloying is a high-energy, solid-state mixing process that combines cold welding and fracture to intimately mix the ceramic to produce a homogeneous distribution of reinforcing Si_3N_4 within the Fe-36Ni matrix. The Si_3N_4 and Fe-36Ni inputs were designed to reach an overall composition of Fe-36Ni + 20vol.%Si_3N_4. The process produces a composite-powder-granule that was then canned, degassed and hotisostatically-pressed (HIP) into square billets. These billets were then forged on a 10MN screw press with a 2:1 forging ratio. Agglomeration of Si_3N_4 was eliminated and a uniform dispersion was achieved, Figure 2.

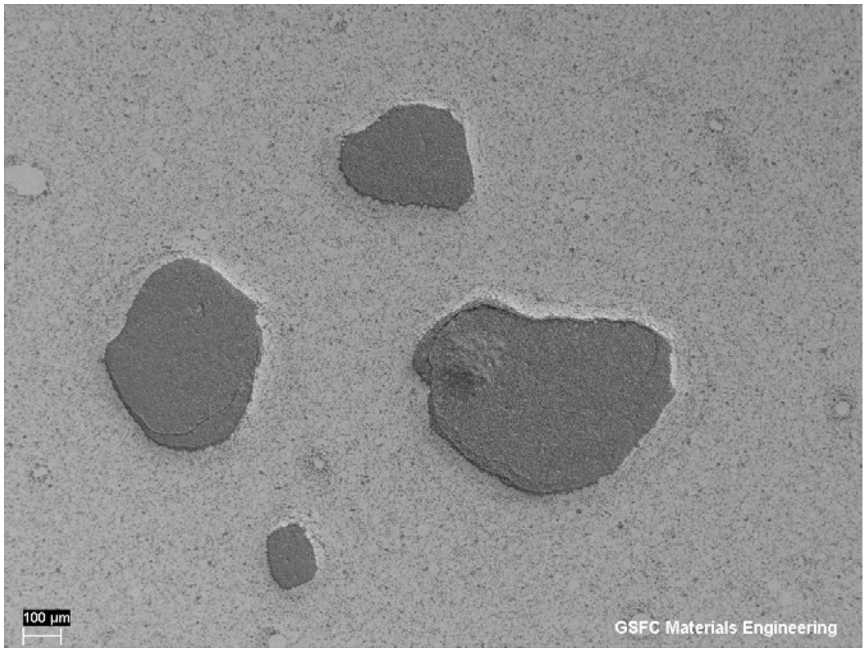

Figure 1. Secondary electron micrograph of twin-cone mixed and HIPed Fe-36Ni+20vol.%Si_3N_4.

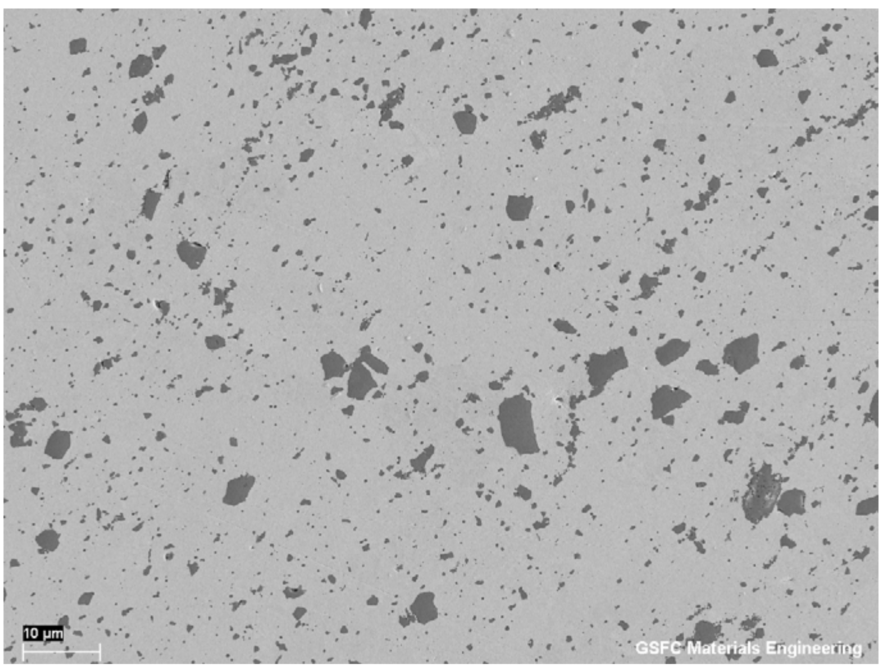

Figure 2. Secondary electron micrograph of MA then HIPed and Forged Fe-36Ni+20vol.%Si_3N_4 (transverse orientation).

Samples were taken from the forged plates and a series of mechanical and physical tests were performed to outline the properties of the new MMC, LoVAR. Tensile properties, thermal expansion and temporal dimensional stability were evaluated.

3. TESTING SUMMARY

Tensile properties

Tensile coupons were cut from the LoVAR forging in the transverse orientation and tested to ISO 6892-1, “Metallic Materials – Tensile testing – Part1: Method of test at Ambient Temperature”. At 100% density, 7.2g/cc, between 4.5-5.0% elongation is observed with yield (0.2% offset) and ultimate tensile strength values at 310MPa and 490MPa respectively. A comparison of stress-strain behavior between conventional wrought Invar® and LoVAR is shown in Figure 3.

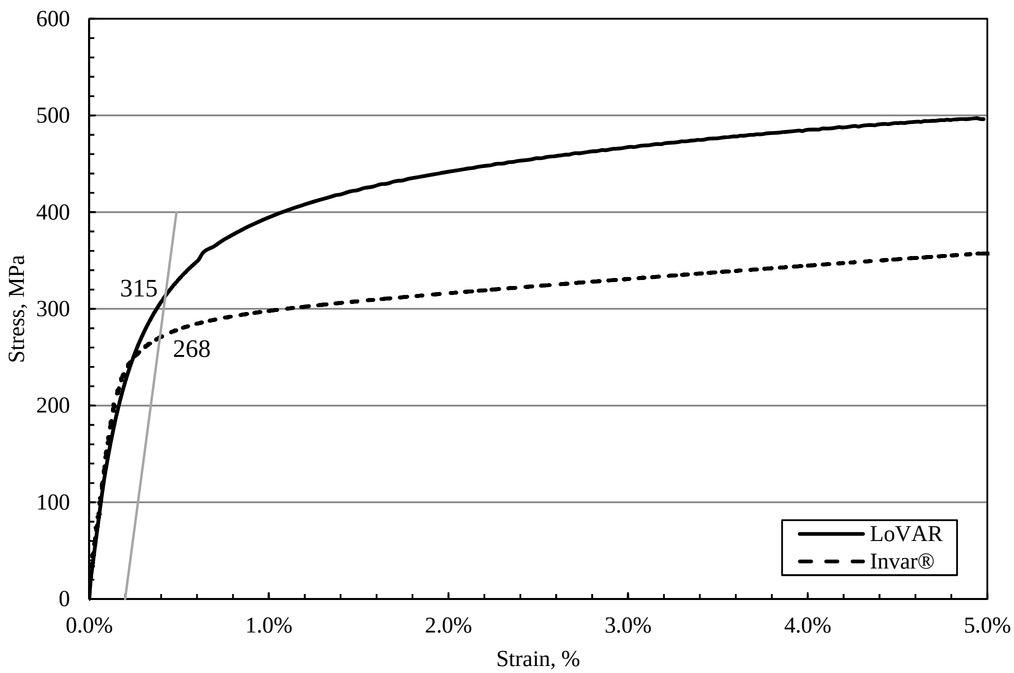

Figure 3. Typical stress-strain behavior LoVAR forging and conventional wrought Invar® with HRB 72 and ASTM grain size 4. Yield at 0.2% offset is indicated on each curve.

Demonstration of five percent ductility makes this early stage development a viable aerospace MMC. Because desired performance directs property requirements, continued experimentation with higher ceramic loadings, HIP, and HIP together with controlled thermomechanical processing for Hall-Petch strengthening are all avenues for development. A fully recrystallized metal matrix ensures isotropic mechanical properties and further simplifies the design of structures for dimensionally stable applications.

Thermal expansion

Heat treatment and chemistry directly affects thermal expansion performance. A variety of heat treatments applied to different Invar® chemistries and their influence on thermal expansion are described in the literature[1]. These are generally three-step treatments beginning with a solutionizing step (785°C – 870ºC) followed by a stress relief (315°C) and concluding with a low temperature treatment (93°C – 100°C) of 48 hours duration. Disorder in the Fe-36Ni matrix, whether produced by heat treatment or plastic deformation, reduces thermal expansion, i.e., reduces CTE[2]. However, plastic deformation also degrades temporal dimensional stability, which is to be avoided[3]. Our work on conventional wrought Invar® shows that the more rapid the cooling rate from the solution treatment temperature, the lower the resulting thermal expansion such that: CTE water quench < CTE gas quench < CTE slow cool, for microstrain measured over a given temperature range[4]. It is speculated that disorder in the lattice site occupancy between the Fe and Ni sites created during the solution treatment is frozen with the more rapid quench and that this disorder contributes to the lower CTE. In addition, we have found the stress relief treatment at 315°C, usually completed with a slow cool, also increases CTE. This may be due to ordering with the Fe and Ni sites during slow cooling through the Curie temperature (280°C). We maintain the concluding 93°C/48 hour treatment because of its beneficial effect on dimensional stability. For manufacturability of LoVAR, chemistry of the Fe-Ni matrix is constrained to be within UNS No. K93603.

Michelson laser interferometry, Figure 4, was used to measure continuous microstrain as a function of temperature.

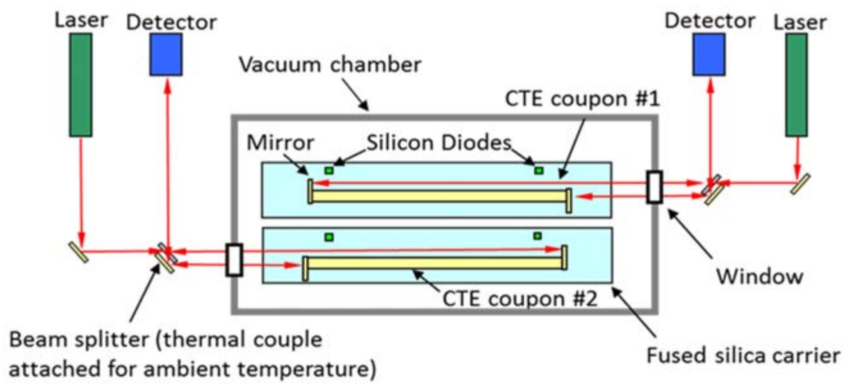

Figure 4. Schematic of Michelson laser interferometer used for measurement of thermal expansion per ASTM E289-04 and isothermal dimensional stability.

The slope of the microstrain curve at a specific temperature provides the instantaneous CTE at that temperature while the slope of the secant between two temperatures is the average CTE between those temperatures. The standard uncertainty in CTE for this measurement is < 0.13 ppm/K. Figure 5 shows the microstrain behavior for the LoVAR forging compared with conventional wrought Invar® and Super-Invar.

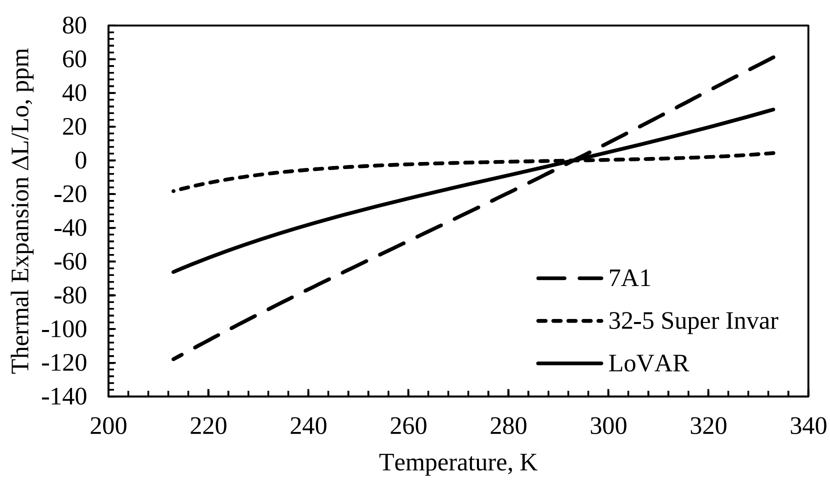

Figure 5. Plot of continuous microstrain as a function of temperature for LoVAR, Super-Invar, and conventional wrought Invar® (labeled as 7A1) Heat treatment is described in text. The slope of each curve at a specific temperature is the instantaneous CTE at that temperature.

LoVAR and the Super-Invar were solution treated in vacuum at 788°C/1 hour, gas quenched in N_2 at 10 bars to ambient and aged at 93°C/48 hours in Ar. The wrought Invar® was solution treated in vacuum at 788°C/1 hour, cooled to ambient at a ramp rate not exceeding 93°C/hour, stress relieved in vacuum at 315°C/1 hour, cooled to ambient with the same ramp rate, then aged at 93°C/48 hours in Ar. From Figure 5 it can be seen that the CTE of LoVAR is less than the CTE of conventional wrought Invar® over the entire measured temperature range. Temperature cycling revealed no hysteresis in the slope of each microstrain curve. Super-Invar is a candidate metal matrix alloy for applications where temperatures are greater than about -55°C as our measurement indicate a martensite start temperature of -79°C. This phase transformation is not shown in Figure 5 as it is just off-scale. A comparison of secant CTE over two temperature ranges is provided in the results section of this paper.

Temporal dimensional stability

One of the major problems using Invar® as a material for optics applications is that it suffers from a time-dependent dimensional change in an isothermal environment. The cause of this property has generated many experiments and a lot of discussion in the literature[5, 6] so it is of interest to evaluate the isothermal dimensional performance of LoVAR.

From the schematic of the laser interferometer in Figure 4, note that part of the laser path length is within the vacuum chamber and is temperature controlled, while another part is outside the chamber and subject to ambient temperature fluctuations. The interferometer measures changes in coupon length by detecting changes in the laser beam path length. As a result, the raw path length measurement is an additive combination of an isothermal part inside the vacuum chamber between the two mirrors on the ends of the coupon, and an ambient part outside the chamber where the room temperature fluctuates. Changes in ambient temperature affect the path length of the laser beam outside the chamber and, hence, the measured length change. Ambient temperature is measured at the beam splitter and changes in path length are subtracted to give the isothermal dimensional behavior. Figure 6 shows the isothermal behavior at 80°C of LoVAR compared with three other samples of conventional wrought Invar® in different heat treat conditions.

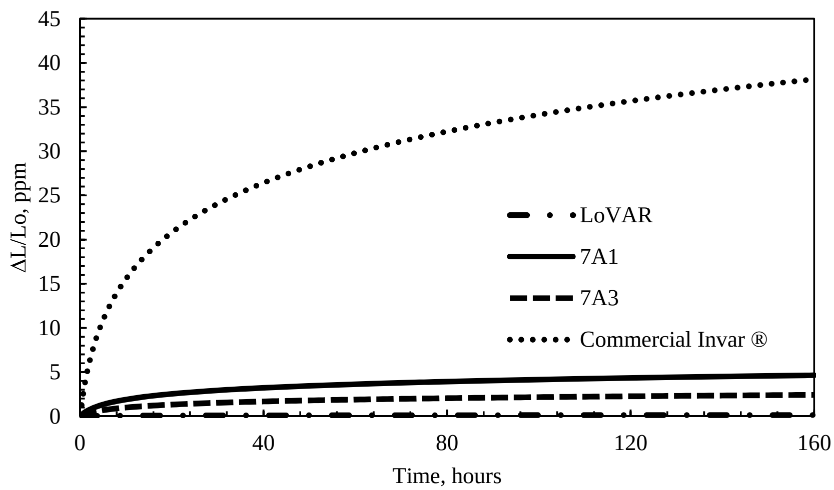

Figure 6. Plot of continuous microstrain over 160 hours at 80°C for LoVAR and three samples of conventional wrought Invar®, each in a different heat treatment condition. Heat treatments are described in the text.

The LoVAR was solution treated in vacuum at 788°C/1 hour, gas quenched in N_2 at 10 bars to ambient and aged at 93°C/48 hours in Ar. The wrought Invar® sample 7A1 was solution treated in vacuum at 788°C/1 hour, cooled to ambient at a ramp rate not exceeding 93°C/hour, stress relieved in vacuum at 315°C/1 hour, cooled to ambient at the same ramp rate, then aged at 93°C/48 hours in Ar. Wrought Invar® sample 7A3, from the same heat as sample 7A1, was solution treated in vacuum at 788°C/1 hour, water quenched, machined to remove scale, then aged at 93°C/48 hours in Ar. The sample labeled commercial Invar® was hot finished rod.

The effect of condition as influenced by heat treatment is apparent in the extent of isothermal dimensional increase in each of the samples. The dimensional performance of LoVAR over 160 hours is nearly flat at this scale. The model fit to these data indicate its dimensional increase is approximately 6% of conventional wrought Invar® processed from 788°C with the water quench. With respect to LoVAR, we speculate that the particle dispersion prevents dislocation glide in the metal matrix, limiting recovery and stabilizing the disorder that provides low CTE and dimensional stability. See Table 3 for values calculated from a model fit to the microstrain plots in Figure 6.

4. RESULTS

Data generated to date during testing of this early-stage development are shown in the tables below.

Table 1. Typical mechanical properties of LoVAR as-forged plate.

Property	LoVAR (Fe-36Ni+20vol.% Si_3N_4)
$R_{p0.2}$ (MPa)	310
R_m (MPa)	490
Elongation (%)	5
Elastic Modulus (GPa)	140
Specific Stiffness (GPa/g/cc)	19.6

Table 2. Thermal expansion performance of LoVAR compared with Super-Invar and conventional wrought Invar®. The LoVAR and Super-Invar were solution treated in vacuum at 788°C/1 hour, gas quenched in N_2 at 10 bars to ambient and aged at 93°C/48 hours in Ar. The wrought Invar® was solution treated in vacuum at 788°C/1 hour, cooled to ambient at a ramp rate not exceeding 93°C/hour, stress relieved in vacuum at 315°C/1 hour, cooled to ambient at the same ramp rate, then aged at 93°C/48 hours in Ar.

Alloy	Room Temperature CTE (ppm/K)	Secant CTE 10˚C to 30˚C (ppm/K)	Secant CTE -60˚C to 60˚C (ppm/K)
LoVAR	0.69	0.69	0.80
Super-Invar	0.05	0.06	0.19
Wrought Invar®	1.49	1.49	1.49

Table 3. Isothermal dimensional stability at 80°C of LoVAR compared with conventional wrought Invar® in three different heat treatment conditions as described in the text. Values calculated from model fit to microstrain plots in Figure 6. Duration of test was 160 hours.

Sample ID	Dimensional change (ppm)
LoVAR	0.14
Invar® (7A1)	4.65
Invar® (7A3)	2.42
Invar® (hot finished rod)	38.16

5. CONCLUSIONS

The mechanical and thermal expansion properties of an early-stage development MMC have been reported. Named, LoVAR, its low CTE and favorable dimensional stability indicates that the ex-situ composite processing paradigm described here produces a structure that enables properties not achievable using any other commercially available metallurgical process. As the first in a new class of dimensionally stable MMCs, the direction is clear for investigating other reinforcing compositions and size distributions to further optimize mechanical and thermal expansion properties. The performance of these new MMCs will find wide application in engineering structures that require dimensional stability in the presence of dynamic thermal disturbances.

6. ACKNOWLEDGEMENTS

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