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NEW HP (HIGH PURITY) INVAR 36*

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Abstract

Rigorous requirements for very low thermal expansivity and temporal instability for metering rods of the Cassini spacecraft Narrow Angle Camera (NAC) were the driving forces at Jet Propulsion Laboratory to develop and qualify for space application a new HP (high purity) Invar 36 material. Conventionally produced Invar 36 could not meet these requirements. High purity was accomplished via powder metallurgy source material. Dimensional stability was a direct result of Invar chemistry and purity and was further enhanced by heat treatment. Experimental test results indicate this material to be the most dimensionally stable Invar 36 ever produced. High purity and thermal treatments ensured both low thermal expansion (CTE from 0.2 to 0.8 ppm/^{0}C) and excellent temporal stability (less than 1 ppm/year) at 38^{0}C and room temperature. In addition, preliminary mechanical test results demonstrated the HP Invar 36 material has similar tensile properties but a higher fatigue endurance limit S_f when compared with conventional Invar 36 (S_f =27 ksi for HP Invar 36 vs. 20 ksi for typical Invar 36). This indicates that HP Invar 36 could also be utilized in loaded precision structure applications. However, microyield strength/microplasticity studies are recommended to fully understand the potential of this material for these applications. HP Invar 36 has already been used for several dimensionally stable critical components of Cassini spacecraft and other space programs as well. These applications, property data and potential commercial usage are described in this paper.

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Introduction

Recent advances in astronomy and high performance requirements forthe next generation precision optical/imaging systems are imposing very stringent demands for dimensional stability of precision structures and science instrument components. Stringent requirements for both very low thermal expansivity and temporal instability for metering rods of the Cassini spacecraft Narrow Angle Camera (NAC) were the driving forces at Jet Propulsion Laboratory (JPL) to develop and qualify for space application a new HP Invar 36 * material. The metering rods had to be made of a material with a coefficient of thermal expansion (CTE) of $< |1|$ ppm/^{0}C and with a combined temporal (long time) stability and thermal hysteresis of $< |1|$ ppm/year.

It was a major challenge to JPL to find a material which could meet these dimensional stability requirements while still possessing other attributes such as mechanical strength and machinability. In the selection process, Invar 36 was chosen as a baseline material because it possibly could meet these requirements through high purity control and appropriate thermomechanical processes. Based on conducted studies (Ref. 1), it was concluded that low carbon/impurirty content notably improves both thermal and temporal stability of Invar 36. In addition, JPL determined that commercially made Invar 36 probably will not meet the (dimensional stability requirements for the metering rods. The commercially Invar alloy production does not have the necessary controls to produce high purity materials and in fact, results in the introduction of impurities during alloying operations. It was decided that the most reliable route to the development of a very stable Invar is ultra-high purity Invar 36. This paper describes how high purity (HP) Invar material was fabricated and procured per JPL instruction. The dimensional stability and mechanical test results are reported and compared with commercially made Invar 36. Finally, current applications in space programs are described and potential commercial usages are envisioned for this unique material.

Fabrication of HP Invar 36 material

HP Invar 36 material was produced by the powder metallurgy process. Powder metallurgy appeared to be a simple and relatively inexpensive manufacturing method necessary to ensure high purity and cleanliness of Invar 36 material. Product purity and tight chemistry-control afforded by the powder metallurgy process could provide reproducible high dimensional stability properties.

During our search, a powder metallurgy manufacturer, Spang Specialty Metals, PA, was contacted and a high purity (HP) Invar 36 sintered billet 10.16 x 10.16 x 137.16 (4" x 4" x 54") was produced per JPL requirements. As part of the manufacturing process control, the elemental powders (Fe and Ni) were thoroughly analyzed and then the alloy was prepared by precise weighing and blending the specified elements. Thus, exact composition and purity were assured. Later, the blended powders were pressed into billet form and alloyed by sintering in a controlled atmosphere. This billet was then sent to Scientific Alloy Inc., Westerly, RI, for thermomechanical processing to further densify and increase the material's strength. Scientific Alloy Inc. cut the billet into two pieces. The first was used to draw the rods of 0.79 cm (0.312") in diameter and 101.6 cm (40") in length. The second was hot hammered into 5.71 cm x 30.48-60.96 cm (2.25" x 10.25" x 12"-24") plate. More manufacturing details are in Ref. 2.

Chemical Composition.

In order to ensure both very good thermal and temporal stability, JPL specified the impurity levels in HP Invar 36 obtained via powder metallurgy. The powders of iron (Fe) and nickel (Ni) should be prepared to a specification such that the total carbon (C) in the final product does not exceed 0.01 percent by weight and that total impurities of manganese (Mn), silicon (Si), phosphorus (P) and aluminum (Al) do not exceed 0.10 percent, preferable each of these impurities should not exceed 0,01 percent individually. This impurity content is extremely critical to dimensional stability of Invar and were held as close to the specification as possible.

The chemical analysis of the rod was performed at JPL and other outside laboratories for comparison study. The results are summarized in Table I below.

Table I. Chemical Analysis Results of HP Invar 36 Rod from Different Labs (in weight percent)

Element	Metals Technology	Atlas Testing	Specialty Alloys	JPL	Desirable Composition
c	0.01	0.005	0.002	0.01	<0.01
Mn	0.01	0.01	<().001	<0.004	<0.01
Si	0.04	0.04		<0.01	<0.01
P	0.005	0.003	<0.01	0.005	<0.01
s	0.005	0.002		0.003	<0.01
Cr	0.01		<0.01		
Al	0.01	<0.01	.<0.001	<0,01	<0.01
Se	0.0001		<0.0001		
Ni	36.24	36.0	36.0	36.8	36.0±0.1
Fe	REM	REM	REM	REM	REM

The analysis results confirmed the high purity of this Invar 36 material. JPL results should be considered as the most reliable in light of the methods employed.

Dimensional Stability

Heat Treatment

The heat treatments of HP Invar 36 rods utilized prior to dimensional stability testing are described in Table 11. The three-step heat treatment was a baseline for evaluation with some other derived treatments.

Measurement Methods

Two kinds of measurements were performed at Optical Sciences Center, University of Arizona:

- Thermal expansion (C1'E/thermal hysteresis - change of lenght with temperature)
- Thermal instability (change of length with time at constant temperature)

Both kinds of measurements relied on the same Fabry-Perot laser- interferometric principle that is described in details in Ref. 3. This method allows us to study the dimensional instability on a daily bask with an accuracy of 0.01 ppm or better (Ref. 4 anti 5). The University of Arizona's Optical Science Center pioneered laser heterodyne and frequency stabilization of lasers and has developed and equipped a laboratory to utilize stabilized laser for precision rneasurernents.

Table II. Heat treatments of HP Invar before dimensional stability testing

ID	Procedure
H.T. # 1	- Annealing at 788 ^{0}C/30 min. slow cool. - Stress relief at 316^{0}C/1 hr. - Aging at 93 ^{0}C/48 hrs.
H.T. # 11	- Specimens were rough machined, annealed and final machined before stress relief and aging per H.T. # 1.
H.T. # 12	- H.T. # 1 + 93 ^{0}C/28.5 days.
H.T. # 2	- Annealing at 788 ^{0}C/30 min., slow cool. - Aging at 93 ^{0}C/96 hrs.
H.T. # 3	- Stress relief at 316^{0}C/48 hrs.

<u>Thermal Stability Test Results</u>.

Coefficient of thermal expansion (CTE)/thermal hysteresis measurements were performed individually for each specimen in the temperature range of -50^{0}C to +50^0 C. All CTE/thermal hysteresis data is briefly summarized in Table 111, In addition, the length changes AL/L vs. temperature were generated to find out how much each specimen failed to return to its original length upon returning to its original temperature (referred to as thermal hysteresis).

Table 111, CTE/Thermal Hysteresis Test Results of HP Invar 36

Condit ion	CTE O°C to 25°C (ppm/^{0}C)	CTE -So"c to +50^{0}C (ppm/^{0}C)	Hysteresis -50^{0}Cto +50^{0}C (ppm/cycle)
As Extruded	0.20	0.29	0.28
H.T. # 1	0.74^a	0.82^a	0.60^a
H.T. # 12	0.75^b	0.81^b	1.60^b
H.T. # 2	0.70	0.81	2.70

[a] mean value of 3 specimens
[b] mean value of 2 specimens

It appears that powder metallurgy made HP Invar 36 behaves similarly to conventionally made Invar 36 in thermal expansivity area, The lowest CTE found in “as extruded” condition indicates that cold working processes increase thermal stability (lower CTE) of HP Invar 36 similar to commercial Invar 36. Also, all stabilization heat treatment with high temperature annealing (at 788°C) increased CTE of HP Invar 36. However, the high purity of HP Invar 36 ensured very low CTE and all specimens, heat treated or as “extruded” had CTE <1 ppm/°C. Although the CTE results for all HP Invar 36 were very consistent, the thermal hysteresis results showed some specimem-to-specimen variation, especially for the heat treated specimens. The CTE values of commercially made Invar 36 found in the JPL previous studies (Ref. 6, 7 and 8)) are shown for comparison studies in Table IV below.

Table IV. CTE of Commercially Made Invar 36 in Room Temperature

CTE (ppm/°C)	Condition
0.8 to 3.0	Depending on chemical composition (purity), H. T. and cold deformation etc.
0.8 to 1.5	Careful chemistry/thermomechanical controls.

The thermal hysteresis data for commercially made Invar 36 was not found in theses studies.

Temporal Stability Test Results.

The temporal stability test was performed for a total of 81 days at a temperature of 38°C. Subsequently the temperature was dropped down to 27 .5°C and length changes were monitored for about 6 weeks. All temporal stability data is summarized in Table V.

Table V. Temporal Stability Test Results of HP Invar 36

Condition	ID	38^0C (ppm/year)	27.5^0C (ppm/year)
As extruded	R1	+2.4	0
	R2	*	*
H.T. # 1	Hi/1	*	*
	Hi/2	*	0
	Hi/3	*	-1.2
Ill'. # 11	H11/1	*	-0.6
	H11/2	-1.0	-0.6
	H11/3	<0	-1.2
H.T.# 12	H12/1	-1.5	*
	H12/2	*	*
H.T. # 2	H2/4	-0.3	*
	H2/2	*	-0.1
	H2/3	-0.8	*
H.T. # 3	H3/1	-1.1	-1.2
	H3/2	-0.8	0
	H3/3	-0.8	-1.0

* questionable results not listed here

It was really difficult to distinguish one particular heat treatment of HP Invar as more effective than any other for temporal changes, Almost all specimens were shrinking no faster than 1 ppm/year at both temperatures. The HP Invar 36 in "as extruded" condition was the most unstable at 38^0C, however, upon dropping to 27.5^0C, it slowed to about zero ppm/year. Also, the temperature change from 38^0C to 27.5^0C, unlike the commercial Invar, did not trigger any drastic new length drift rate for the heat treated specimens. The temporal stability values of commercially made Invar 36 are shown for comparison studies in '1'able VII below.

Table VI. Temporal Stability of Commercially-made Invar 36

Temporal Stability	Condition
up to +11 pprn/day	For various Invar composition and thermomechanical conditions at 20 to 70°C (Ref. 9, 10 and 11)
+1.5 to +27 ppm/year	University of Arizona's studies at up to 60°C.

Mechanical Properties

Tensile.

Tensile properties of HP Invar 36 are shown in Table VII. The HP Invar 36 was three-step heat treated before the testing. Some typical tensile properties of commercial-made Invar 36 in the annealed condition are listed here as well.

Table VIII. Tensile Properties of HP Invar 36 and Commercially-made Invar 36 (Ref. 12)

	HP Invar 36		Invar 36
	Rod	Plate	Typical
Tensile Strength, ksi:			
L	67.8	67.6	71.0
T		66.2	
Yield Strength, ksi:			
L	39.3	39.2	40.0
T		38.5	
Elongation, %:			
L	70	67	41
T		63	

L - mean value in longitudinal direction
T - mean value in transverse direction

Tensile test results of HP Invar 36 indicate similar tensile strength but much better ductility (elongation) than the conventional Invar 36.

Fatigue

Fully reversed axial fatigue test results of HP Invar 36 at room temperature are presented in Figure

1. The specimens were taken from a plate in longitudinal direction and heat treated (annealed) before testing,

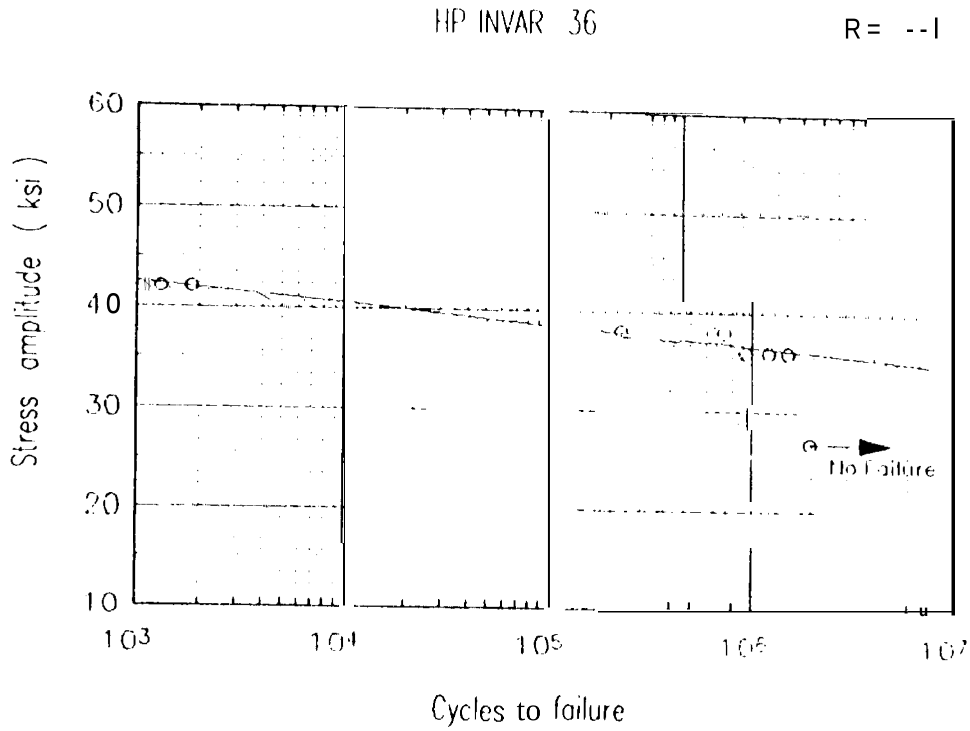

Figure 1 Axial fatigue behavior of HP Invar 36 at room temperature

The axial fatigue data of smooth 0.10 cm (0,040") commercially-made Invar 36 sheet with some cold work is shown for comparison studies in Fig. 2 (Ref. 12).

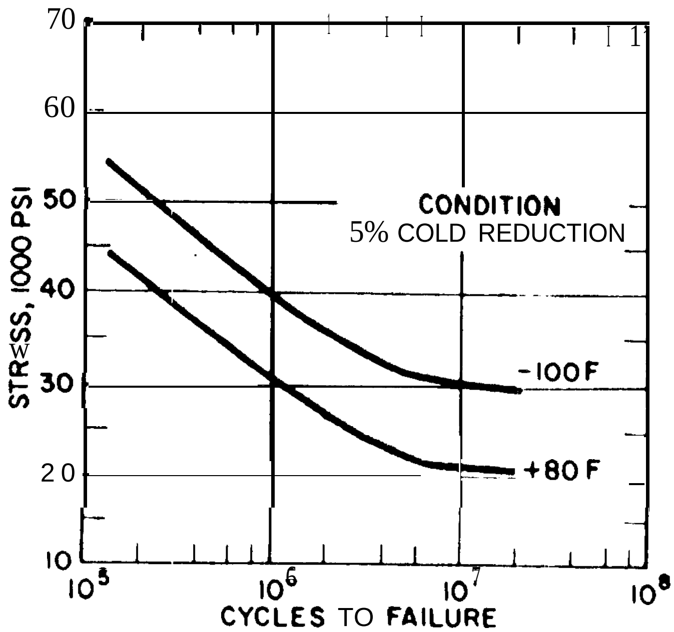

Figure 2 Axial fatigue characteristics of Invar 36 sheet at different temperatures.

It appears that HP Invar 36 plate is super-ior at least in a high-cycle fatigue region (>10^6 cycles) when compared with commercially made Invar 36 sheet at room temperature. The endurance limit for'commercially made nvar 36 sheet is - 20 ksi vs. 27 ksi or higher for HP Invar 36 plate.

Summary

JPL has succeeded in obtaining possibly the most dimensionally stable Invar 36 material ever produced called HP Invar 36* or "JPL Invar." High purity and cleanliness of HP Invar 36 attained by the powder metallurgy process, ensured both low thermal expansion and excellent temporal stability. CTE <11 | ppm/°C along with temporal stability <11 | ppm/year were achieved together. Stabilization heat treatment increased the CTE (from 0.2 ppm/°C to approx. 0.8 ppm/°C) but it was really difficult to distinguish any particular heat treatment as more effective than any other for temporal stability, Almost all specimens were shrinking slower than 1 pprn/year. It appears the three-step heat treatment with annealing after rough machining (H.T. # 11) had the lowest thermal hysteresis among the heats treated HP Invar 36 specimens with low thermal expansion. and good temporal stability as well.

These dimensional stability characteristics have never been reported before for any Invar material. Although low thermal expansion (CTE <11 | ppm/°C) had been reported, the temporal stability was sacrificed or not measured at that time. Previously reported low CTE's had achieved by cold working, fast cooling during heat treatment and other thermomechanical methods which introduced temporal instabilities in Invar 36 materials. Although internal/quenching stresses could be reduced by the stress relief heat treatment, the expansion change is the carbon-dependent phenomenon (Ref. X) and sometimes takes many years of natural and artificial (elevated temperature) aging to stabilize Invar 36 material with high carbon and other impurities content.

HP Invar 36 has already been used for some dimensionally stable critical components of Cassini spacecraft and other space programs as well. In addition to space applications, the HP Invar 36 could be used in commercial sectors in such applications as in low-expansion mandrels, molds for use in production of low-expansion polymeric composite parts, stable optical mounts, support assemblies for commercial laser interferometers, cameras, watch industry, microelectronic packaging, stable mechanical support parts for precision devices such as computer disc drives, read/write mechanisms, profilometers, commercial satellite instrumentation and other applications in areas of metrology, instrumentation and precision optical systems.

However, 11P Invar 36 has not been used so far for loaded precision structures because of lack of the microyield strength data. Preliminary mechanical test results demonstrated the HP Invar 36 material has similar tensile strength, higher ductility and fatigue endurance limit when compared with the conventional Invar 36, This indicates HP Invar could be also utilized in loaded precision structure applications although microyield strength/microplasticity studies are recommended to fully understand the potential of this material for these applications. As of this writing, the HP Invar 36 material is in process of testing and evaluating the microyield strength at Research and Development Center, Lockheed Missiles & Space Co, Palo Alto, CA. If the microyield strength will be higher than in conventional Invar 36, then 11P Invar 36 could have the optimum combination of dimensional stability (thermal and temporal), tensile strength, dynamic load resistance and microyield strength. Discovered 100 years ago, the conventionally made Invar 36 has some limitations especially in such areas as temporal stability and resistance to dynamic loads. On the other hand, HP Invar could eliminate these drawbacks and be a basic material for a myriad of potential applications in space and commercial sectors.

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