

## Dimensional stability of High Purity Invar 36

Witold Sokolowski, Stephen Jacobs\*, Marc Lane, Tim O'Donnell and Cheng Hsieh

Mechanical Systems Engineering and Research Division  
Jet Propulsion Laboratory  
California Institute of Technology  
Pasadena, CA 91109

### ABSTRACT

High performance requirements for the Imaging Science Subsystem (ISS)/Narrow Angle Camera (NAC) instrument on the NASA/Jet Propulsion Laboratory (JPL) Cassini spacecraft impose very stringent demands for dimensional stability of metering rods in the camera's athermalizing system. Invar 36 was chosen as a baseline material because it possibly could meet these requirements through high purity control and appropriate thermomechanical processes. A powder metallurgy process appears to be the manufacturing method to ensure high purity and cleanliness of this material. Therefore, a powder metallurgy manufacturer was contacted and high purity (HP) Invar 36 was produced per JPL engineering requirements. Several heat treatments were established and heat treated 11P Invar 36 samples were evaluated. Coefficient of thermal expansion (CTE), thermal hysteresis and temporal stability test results are reported here. The test results indicate that JPL has succeeded in obtaining possibly the most dimensionally stable (lowest CTE plus lowest temporal change) Invar 36 material ever produced. CTE < 1 ppm/°C are reported here along with temporal stability < 1 ppm/year. These dimensional stability properties will meet the requirements for metering rods on the Narrow Angle Camera.

### 1. INTRODUCTION

The Jet Propulsion Laboratory (JPL) is presently building and testing a near ultraviolet and visible wavelength Imaging Science Subsystem (ISS)/Narrow Angle Camera (NAC) instrument (Fig. 1) for use on the Saturn-bound NASA/JPL Cassini spacecraft. This imaging system is a next generation system compared to those which JPL has flown on previous spacecraft. The optical prescription is more demanding of tight tolerances as optical performance goals of broader spectral range are much more ambitious while maintaining the diffraction limited system of the Voyager spacecraft design. For bulk temperature changes, the camera has an "athermalizing system". This system uses the metering rods which allow the optical elements to move relative to the camera and to themselves in such a way as to remain in their optimal positions as the temperature of the system varies. However, the elimination of aperture correctors and the use of aspheric surfaces on the mirrors makes the design very sensitive to dimensional errors which in turn impose very stringent demands for dimensional stability of the metering rods in the athermalizing system. In order to meet the camera's optical requirements, the rods should meet rigorous requirements for very low thermal expansivity and temporal instability, possibly at a magnitude never required before. The metering rods must be made of a material with a coefficient of thermal expansion (CTE) of < 1 ppm/°C and with combined temporal (long term) stability and thermal hysteresis (return to original length after thermal cycling) 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 necessary attributes such as mechanical strength and machinability. In the selection process, Invar 36 material was chosen as the baseline material. In previous JPL missions such as the Viking and Galileo spacecraft, conventionally produced Invar 36 was used for metering rods and it worked well enough. However, dimensional stability requirements were not as rigorous as they are for this next generation ISS/NAC imaging system instrument. According to our studies, the Invar 36 material could possibly meet the dimensional stability requirements if high purity control and appropriate thermomechanical processes were maintained. Super-Invar (Fe-Co-Ni), although superb at room temperature, was eliminated during the selection process for several reasons discussed later in this paper.

\*Optical Sciences Center, University of Arizona

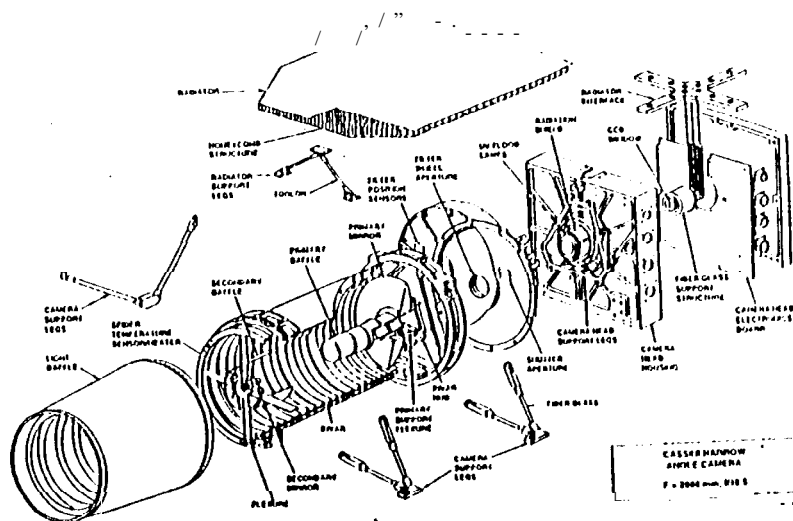


Figure 1: Current Configuration of Narrow Angle Camera (NAC for Cassini Spacecraft

This paper describes how JPL determined the requirements for Invar 36 material (chemical composition, manufacturing and process methods, etc.) which possibly could meet the dimensional stability requirements for ISS/NAC metering rods. It also reports how high purity (1 IP) Invar was fabricated and procured per JPL instructions, heat treated in order to improve dimensional stability even further and finally tested. As a result, JPL has succeeded in obtaining possibly the most dimensionally stable Invar 36 material ever produced.

## 2. DIMENSIONAL STABILITY OF INVAV 36: PREVIOUS STUDIES

Since its discovery by Guillaume (Ref. 1) in 1896, Invar (Fe-36Ni) has proved of considerable scientific and practical interest because of its remarkably low coefficient of thermal expansion (CTE) near room temperature. This effect is attributed to the unique magnetic feature of this alloy (Ref. 2). Depending upon chemical composition, heat treatment, extent of cold deformation and other subtle factors, the CTE of Invar has been reported (Ref. 2- 8) to vary from  $-0.610 + 3.0 \text{ ppm}/^\circ\text{C}$  in the temperature range at  $-70$  to  $+ 100^\circ\text{C}$ . With careful controls, it is commercially practical to produce Invar having less-variable CTE values, e.g.,  $0.8101.6 \text{ ppm}/^\circ\text{C}$  in the range of  $30$  to  $100^\circ\text{C}$  (Ref. 9). In general, the CTE of Invar is markedly influenced by carbon and other impurities and typically increases with increasing carbon/impurities content. The CTE of Invar is also influenced by thermal and mechanical treatments. For example, fast water quenching from about  $800^\circ\text{C}$  produces a lower CTE than does furnace cooling. The CTE is further reduced by cold working and even negative values have been reported (Ref. 8).

It is not generally known among Invar's users that its excellent thermal stability (low CTE) is not necessarily matched by its isothermal temporal (long term) stability. Temporal instability values as high as  $+ 11.0 \text{ ppm}/\text{day}$  at temperatures of  $20$  to  $70^\circ\text{C}$  have been reported for various Invar composition and thermomechanical condition (Ref. 3, 5, 6, 7, 10, 11 and 12). Unfortunately, correlation of some publicized data was very difficult because of the wide variations and often incomplete records of test methods, measurement sensitivity, material composition and thermomechanical anti testing conditions.

Laser measurement technologies have facilitated tremendous progress in measurement sensitivity and have made it much easier to study dimensional instability on a daily basis with accuracies of  $0.01 \text{ ppm}$  or better (Ref. 13- 17). The University of Arizona's (U of A) Optical Science Center pioneered laser optical heterodyne and frequency stabilization of lasers and has developed and equipped a laboratory to utilize stabilized lasers for precision measurements (Ref. 18- 22). Very significant publications on the topic of Invar and Super-Invar's temporal stability

have come from use of this laboratory. All these studies, performed on commercial Invars, indicated the large effect of carbon and other impurities on temporal stability properties. Temporal stability increases (lower length change rate) with decreased carbon and impurities content. Temporal stability is also influenced by test temperature, temperature changes, heat treatment and thermomechanical processes. In general, Invar 36 tested at an elevated temperature up to 60°C had lower temporal stability than at room temperature (at least for several months during testing) and temperature changes caused a difference in length change rates. The heat treatment, depending on previous Invar process history, may increase or decrease the temporal stability. Sometimes the simplest treatment could be the most effective (Ref. 18). The main purpose of the heat treatment is to stabilize/stress relieve Invar 36 material and accelerate the aging process. In U of A's studies, the commercial Invar 36 expanded, with rates varying from 1.5 to 27 ppm/year. All these findings are consistent with the finding of Lement et. al., in 1950 (Ref. 3). In this landmark paper, the Massachusetts Institute of Technology (MIT) team proposed the mechanism of Invar dimensional instability. It appears, according to this theory, that in most commercial Invar 36 investigated (with some carbon content), the carbon-dependent  $\gamma$ -expansion was the dominant phenomenon causing Invar to expand with time. Also Super-Invar (Fe-Co-Ni) was investigated at the U of A's Optical Science Center. This material, although superb at room temperature, was eliminated by JPL during the selection process because of its highly composition-dependent irreversible phase transformation, temperature-dependent temporal stability and difficulties in fabrication.

Based on conducted studies, described above, it was concluded that low carbon/impurity content notably improves both thermal and temporal stability of Invar 36. In addition, JPL determined that commercially made Invar 36, very likely will not meet dimensional stability requirements for metering rods and the most reliable route to development of a very stable Invar is ultra-high purity Invar 36. It was suggested that a very pure Invar 36 could have both thermal and temporal properties at least as good as Super-Invar without the problems just mentioned above.

### 3. HIGH PURITY (HP) INVAR 36 MATERIAL

A powder metallurgy process 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. In the powder metallurgy process, accurate amounts of pure elemental powders are weighed to meet the exact chemical composition. The blended powders are pressed into billet form and alloyed by sintering in a controlled atmosphere. In conventional alloy production, such as air or vacuum melting, there are two major sources of contamination which sometimes cannot be eliminated: deoxidants added during the melting operation and refractory materials picked up in the furnace during melting and from the mold lining when the metal is poured. Limited chemistry control of conventionally melted products is generally "during the event". A sample analysis is made during the melting and then adjustments are made accordingly. The last analysis is reported as the alloy chemistry but may not represent the actual composition throughout the melt. On the other hand, powder metallurgy products are controlled "before the event". Prior to use, the elemental powders are thoroughly analyzed and then the alloy is prepared by precise weighing and blending the specified elements. Thus, exact composition and purity are generally assured.

During our search, a powder metallurgy manufacturer, Spang Specialty Metals, Butler, PA, was contacted and a high purity (111') Invar 36 sintered billet 10.16 cm x 10.16 cm x 137.16 cm (4" x 4" x 54") was produced per the JPL requirements. This billet was then sent to Scientific Alloy, Inc., Westerly, R.I., for further thermomechanical processes to increase the material's strength and density. Scientific Alloy, Inc., cut the billet into two pieces. The first was used to draw (extruded) the rods of 0.79 cm (.312") diameter and 101.6 cm (40") length. The second was hot hammered into 5.71 cm x 26.03 cm x 30.48-60.96 cm (2.25" x 10.25" x 12"-24") slabs. The extruded rods were planned to be used for the ISS/NAC metering rod and were tested for dimensional stability later on. Chemical analysis, performed at JPL, and at other labs for comparison study, confirmed the high purity of this Invar 36 material, with a carbon content of 0.01% or less and other impurities less than 0.01% (Table I).

### 4. TEST PLAN FOR DIMENSIONAL STABILITY EVALUATION

A test plan for dimensional stability evaluation was designed based on the assumption that the findings from previous studies on commercial Invars could be also applied to HP Invar 36. This test plan was described in Ref. 23 and is briefly summarized in Table 11. The proposed dimensional stability evaluation activities of 111' Invar 36

included several types of heat treatment before testing. These heat treatments were to minimize the temporal length changes by a stress relieving operation and accelerating the aging process before HIP Invar 36 is finished to size and placed in service. The heat treatments increase temporal stability but sometimes CTE is sacrificed. In addition to CTE and temporal stability testing, thermal hysteresis testing was performed in order to find out the effect of thermal cycling expected during the Cassini Mission. Therefore, the heat treatment to yield the best temporal stability/CTE/thermal hysteresis was to be selected for flight use.

It was planned to perform the CTE/thermal hysteresis measurements individually for each specimen within the temperature range of -50°C to +50°C in 25°C increments and the overall CTE was to be determined. The temporal stability test was planned to be performed for at least 60 days at a temperature of 38°C because most dimensional instabilities were expected to occur at elevated temperatures during the first two months of testing.

## S. MEASUREMENT METHODS

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

1. Thermal expansion (CTE/thermal hysteresis - change of length with temperature), and
2. Temporal instability (change of length with time at constant temperature).

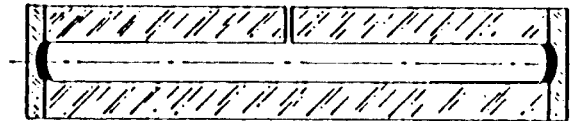


Figure 2. Typical Sample/Vitalon Configuration.  
Confocal dielectric coated mirrors are held in place by gravity.

Both kinds of measurements rely on the same laser-interferometric principle. The sample is configured to form the spacer between two concave mirrors, thereby forming a confocal Fabry-Perot resonator (Fig. 2). A tunable HeNe laser is optically aligned with this resonator, the cavity resonant frequency is observed, and the laser is then locked to the cavity resonant frequency. Lastly, a record is made of this laser frequency with respect to a stabilized reference laser. If at some later time the sample length changes due to time or temperature changes, then the cavity resonances change by an amount  $\Delta\nu$ . This frequency shift is measured by relocking the tunable laser to the new cavity resonant frequency and again comparing the laser frequency with respect to the reference laser. In this way, we obtain an absolute measure of sample length change through the relation.

$$\frac{\Delta L}{L} = \frac{\Delta\nu}{\nu}$$

A shift  $\Delta\nu=474 \text{ MHz}$  corresponds to 1 ppm.

Fig. 3 shows the experimental arrangement used for CTE/thermal hysteresis measurements. A sample was oriented vertical with optical axis in a vacuum better than .01 Torr. The AI. and temperature data were recorded only after sample length stabilized to  $\Delta L/L, < .001 \text{ ppm/hour}$ . CTE/thermal hysteresis measurements were performed individually for each specimen in the temperature range of -50°C to +50°C, stopping every 25°C to record  $\Delta\nu$  and temperature. Plots were made of frequency shift vs. temperature, which were converted to  $\Delta L/L$  vs. temperature. This also showed in detail how much each sample failed to return to its original length upon returning to its original temperature (referred here as thermal hysteresis).

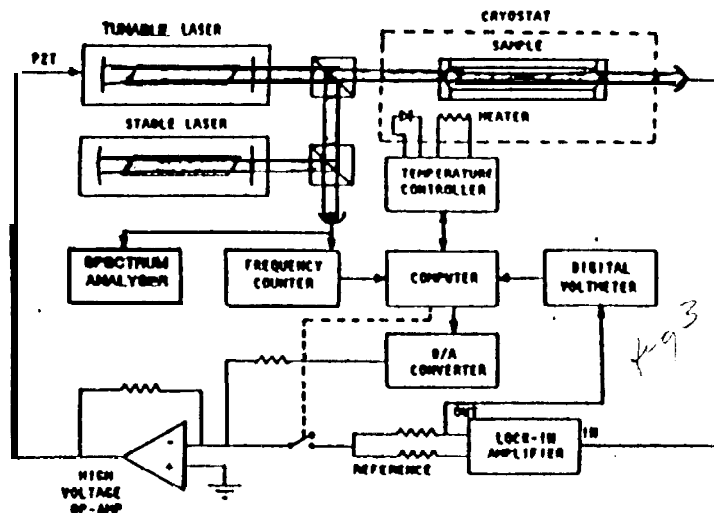


Figure 3. Two-laser arrangement used to measure thermal expansion.

Fig. 4 shows the arrangement used for temporal stability measurements. The massive copper sample holder had a capacity of 37 samples: 35 were supplied by JPL (including some other dimensionally stable, nonmagnetic candidate materials), and two were supplied by the University of Arizona - a copper sample (used for temperature stabilization) and an optically contacted Homosil sample (used as a fused silica double check on the stability of the reference laser). Temporal stability testing was performed at 38°C for over 11 weeks (80 days), after which the chamber temperature was dropped down to ambient (27.5°C) and the specimen's length changes were monitored for another 6 weeks (43 days). The copper reference sample indicated the chamber temperature was held constant to  $\pm .015^\circ\text{C}$ . The Homosil reference sample remained constant in length with  $\pm .01\text{PPM}$ , indicating that the stable reference laser was indeed stable to  $10^{-8}$  over the test duration. Each weekday a measurement was made, sequentially, of initial chamber temperature, each sample's resonant frequency change and final chamber temperature. These resonant frequency changes were plotted vs. time and later converted to  $\Delta L/L$  vs. time.

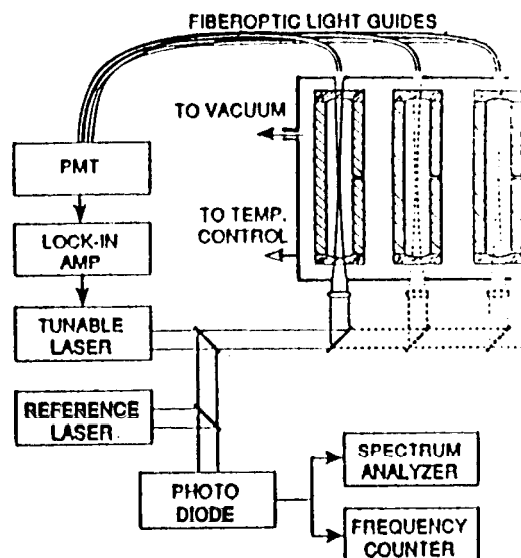


Figure 4. Arrangement used to study changes of length at constant temperature.

## 6. TEST RESULTS

### 6.1. CTE/thermal hysteresis

All CTE/thermal hysteresis data is briefly summarized in Table 111, Typical curve length change vs. temperature (sample R3) is presented in Fig. 5. Specimen diameter of 0.76 cm was used. In addition to overall CTE within the temperature range of  $-50^{\circ}\text{C}$  to  $+50^{\circ}\text{C}$ , the CTE within the temperature range of  $0^{\circ}\text{C}$  to  $25^{\circ}\text{C}$  was also determined for each material and shown in Table III. The metering rods are expected to see this latter temperature range most of the time during the Cassini mission,

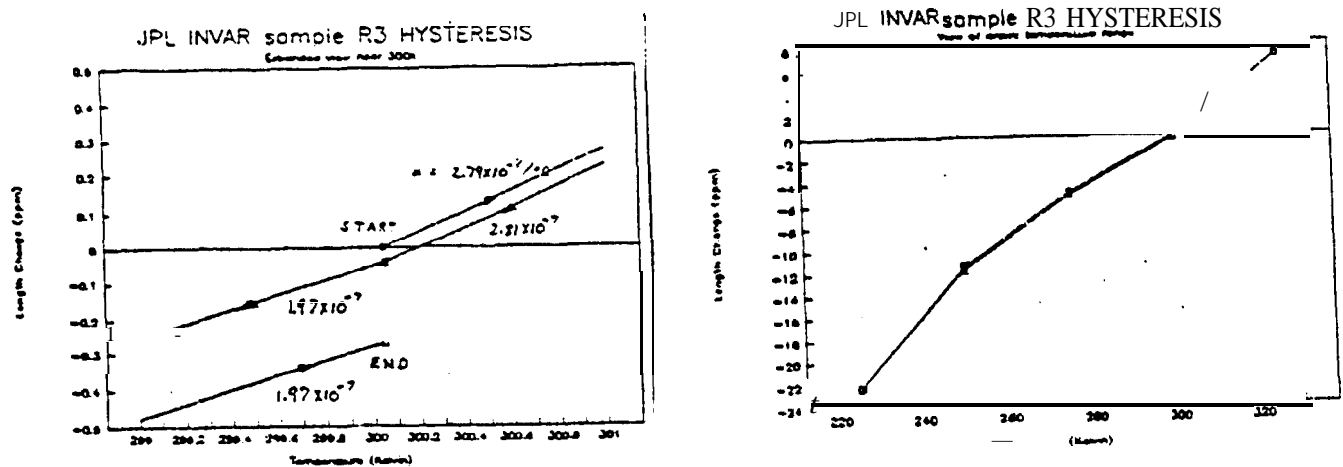


Figure 5. Typical CTE/hysteresis data. "As received" Sample R3

It should be noted that several machined and heat treated IIP Invar 36 specimens were bent (possibly due to machining) to the extent that CTE measurements were impossible, despite numerous attempts. One of the bent temporal stability specimens (1-12/1") was replaced by a less-bent specimen (1-12/4\*), which was barely usable for CTE testing. Furthermore, the 111' Invar 36 CTE specimens from the 11.1' #3 pool, such as 113/4\*, 113/5\* and 113/6\*, were bent excessively and were not tested at all.

The CTE test results indicated that among the IIP Invar 36 specimens measured, both the lowest CTE and the lowest thermal hysteresis was found for samples in the "as extruded" condition. All heat treatments, except I-I-T #3 which was not tested for CTE, increased CTE of HP Invar 36 (almost four times from  $0.2 \text{ ppm}/^{\circ}\text{C}$  to approximately  $0.8 \text{ ppm}/^{\circ}\text{C}$ ). The extent of cold-work in IIP Invar 36 rods in "as extruded condition was apparently sufficient to reduce the CTE markedly at these test temperatures. However, all IIP Invar 36 specimens ("as extruded" and heat treated) meet the thermal expansion requirements for metering rods of  $\text{CTE} \leq 1 \text{ ppm}/^{\circ}\text{C}$ . Although the CTE results for all IIP Invar 36 specimens are very consistent, the thermal hysteresis results showed some specimen-to-specimen variation, especially for the heat treated specimens. Hysteresis values ranged from 0.12 to 2.70 ppm/cycle.

### 6.2. Temporal stability

1-hc temporal stability test was performed for a total of 81 days at a temperature of  $38^{\circ}\text{C}$ . Subsequently the temperature was dropped down to ambient ( $27.5^{\circ}\text{C}$ ) and length changes were monitored for about 6 weeks. Due to cost considerations, all sample mirrors were held in place by gravity, rather than by optical contacting (previous U of A study concluded the optical contacts were not essential, especially for usual larger 2.54 to 3.17 cm diameter

"See Table I

specimens and mirrors). However, the 1-11' Invar 36 specimens had small diameters (about 0.76 cm), which necessitated use of small (and light weight) mirrors to form optical resonators. These small mirrors were susceptible to jumps and settlements caused by vibrations and perhaps dirt specks and/or electrostatic forces. Fortunately, we had more than one specimen of each type which made it possible to draw conclusions, although with varying certainty.

All temporal stability data is summarized in Table IV. Typical temporal stability data (sample H12 #2) is shown in Fig. 6. The length change rates represent the slope of the linear portion of each  $\Delta L/L$  vs. days, U of A has fitted straight lines to the data. A least square analysis was not done because other uncertainties seemed far greater. In U of A judgment, all the nonlinear data should be considered questionable with the best possible conclusion drawn from the linear segment. It was not clear how to interpret and qualify the data in cases where the length changed in a nonlinear manner. The cause of the nonlinearity could have been material relaxation but U of A suspected it was something like a speck of dirt or electrostatic charge repulsion. JPL analyzed the temporal stability test results fitting exponential lines to the data. It was concluded that the U of A interpretation represented conservative, i.e., higher length change rate values when compared to the calculated rates where the nonlinear data is considered,

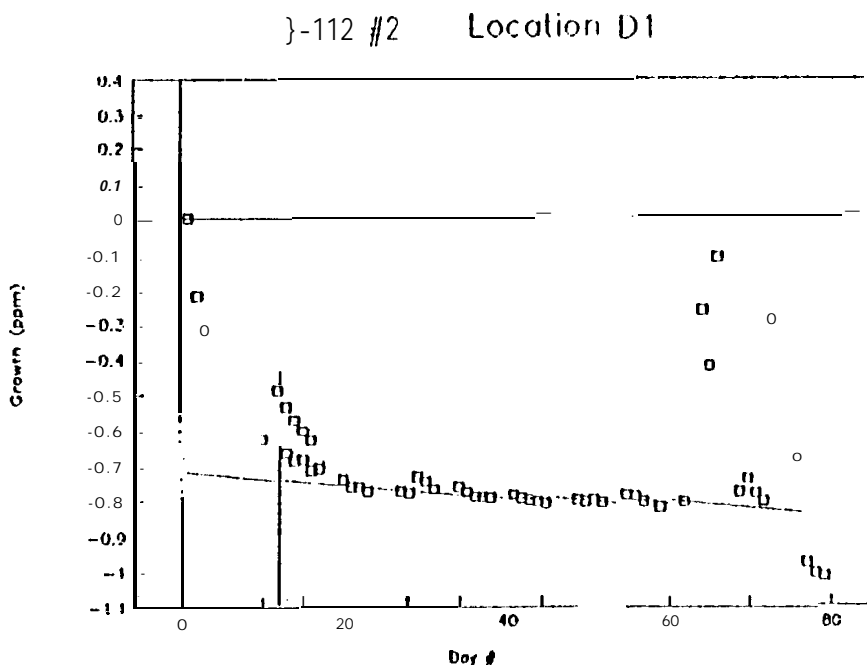


Figure 6. Typical temporal stability data heat treated Sample H12 #2

The dimensional stability effectiveness provided by any particular heat treatment of HP Invar 36 was not clear. Almost all specimens were shrinking no faster than 1 ppm~/year. The samples H.T. #1 gave the most nonlinear results at 38°C, while the 1-1. T. #3 provided the confident straight line data. The H.P. Invar 36 in "as extruded" condition was the most unstable at 38°C for eleven weeks, however, upon dropping to 27.5°C for six weeks, it slowed to about zero ppm~/year. These eleven weeks at 38°C could be a good stabilizing treatment for HP Invar 36 to be used at ambient temperature. However, since the temporal stability of two specimens were so different, there are residual concerns. The temperature change from 38°C to 27.5°C did not trigger any drastic new length drift rate for the heat treated HP Invar specimens. In general, it improved dimensional stability and provided more confident straight line data for three-step heat treatments H.T. #1 and H.T. #11 while a simple two-step stress relief/stabilizing heat treatment H.T. #3 was not much affected and gave straight line data and good temporal stability at both temperatures.

## 7. DISCUSSION

The dimensional stability test results reported here confirmed our original hypothesis: high purity (HP) Invar 36 made by powder metallurgy is an exceptionally dimensional stable material. It was proven that high purity (carbon and other impurities content  $<0.01\%$ ) ensured both low thermal expansion and excellent temporal stability.  $CTE < 1$  ppm/ $^{\circ}C$  along with temporal stability  $< 1$  ppm/year were achieved together. These dimensional stability characteristics have never been reported before for any Invar material. Although low thermal expansion ( $CTE < 1$  ppm/ $^{\circ}C$ ) had been reported before, the temporal stability was sacrificed or not measured at that time. Previously reported low CTE's had been achieved by cold working, fast cooling during heat treatment and other thermomechanical methods which introduced temporal instabilities in Invar 36 materials. Although internal/quenched stresses could be reduced by the stress relief heat treatment, the expansion change is the carbon-dependent phenomenon (Ref. 3) and sometimes takes many years of natural and artificial (elevated temperature) aging to stabilize Invar 36 material with high carbon and other impurities content. Also, the temperature change could trigger new length drift rate for already stabilized commercial Invar 36.

According to the Lement theory (Ref. 3), its high purity and extremely low carbon content of HP Invar 36 eliminated the expansion with time ( $\gamma$ -expansion phenomenon) caused by redistribution of the carbon atoms in solid solution. Actually, all HP Invar 36 specimens with or without the heat treatments were shrinking very slightly at  $27.5^{\circ}C$  and at  $38^{\circ}C$  as well during temporal stability testing. It could be interpreted (using above mentioned theory) that this contraction was possibly due to the relief of internal stresses left after the thermomechanical processes (e.g., extrusion)/ $\gamma$ -cat treatments. It appears that powder metallurgy made HP Invar 36 behaves in part similarly to conventionally made Invar 36 especially in thermal expansivity area. The lowest CTE found in "as extruded" condition indicates that cold working and other thermomechanical processes increase thermal stability of HP Invar 36 similar to commercial Invar 36. Also, all stabilization heat treatments with high temperature annealing (at  $788^{\circ}C$ ) increased CTE of HP Invar 36. However, all HP Invar 36 specimens ("as extruded" and heat treated) had low CTE of  $< 1$  ppm/ $^{\circ}C$  which suggests the high purity of HP Invar 36 ensured CTE low enough for all specimens to meet thermal stability requirements for the ISS/NAC metering rods.

Regarding long-term stability area, it is really difficult to distinguish any particular heat treatment of HP Invar 36 as more effective than any other for temporal changes. Almost all specimens were shrinking no faster than 1 ppm/year at both temperatures, which is a noteworthy achievement and meets temporal stability requirements for metering rods. Also, the temperature change from  $38^{\circ}C$  to  $27.5^{\circ}C$ , unlike sometimes in commercial Invars, did not trigger any drastic new length drift rate for the heat treated HP Invar 36 specimens. Furthermore, all heat treatments could meet both thermal and temporal stability requirements but thermal hysteresis results showed some specimen-to-specimen variation for the heat treated specimens. Although the lowest hysteresis results were for "as extruded" condition, it is suggested to test more specimens in each condition to have more conclusive results. The simple low temperature, two-step stress relief/stabilizing heat treatment (I.T. # 3) did not result in any significant stability variation with temperature change and provided more confident linear, temporal stability data at both temperatures which is in agreement with some findings with conventionally made Invar 36 (Ref. 18). Unfortunately, the I.T. # 3 specimens were not tested for CTE/thermal hysteresis for some reasons described above. Based on previous studies, we can assume only the CTE values could be lower than for the high temperature, three-step heat treatments but we have no basis to make any assumptions regarding thermal hysteresis. The three-step heat treatment I.T. # 11 appears to have the lowest thermal hysteresis among the heat treated HP Invar 36 specimens with low thermal expansion and good temporal stability as well.

All the  $27.5^{\circ}C$  data showed excellent temporal stability, but we have no way to be sure what would have happened without the previous eleven weeks  $38^{\circ}C$  exposure. One cannot escape regarding the eleven weeks at  $38^{\circ}C$  as part of the heat treatment. Then, the assured conclusion for the lowest CTE, lowest hysteresis and the best temporal stability at ambient temperature ( $27.5^{\circ}C$ ) could be HP Invar 36 in "as extruded" condition with 11 weeks stabilization treatment at  $38^{\circ}C$ . However, this HP Invar 36 was very unstable (high length change rate) at  $38^{\circ}C$  and temporal stability of two specimens at  $27.5^{\circ}C$  were not consistent, which may cause some concerns. Perhaps longer time of aging treatment at  $38^{\circ}C$  is needed for complete stabilization and very good temporal stability of "as extruded" at ambient temperature.



## 8. CONCLUSIONS

The following summarizes the dimensional stability test results of our high purity **HP Invar 36**. Despite some uncertainties in data interpretation of temporal stability test results, numerous points are clear here:

- <sup>0</sup>JPL has succeeded in obtaining possibly the most dimensionally stable **Invar 36** material ever produced (sometimes called **HP Invar 36** or "**JPL Invar**").
- <sup>0</sup>High purity and cleanliness of **HP Invar 36**, i.e., carbon content <01% and other impurities <.01% ensured both low thermal expansion and very good temporal stability.
- <sup>0</sup>Almost all I-1P **Invar 36** specimens wet-c exceedingly stable with time ( $c 1 \text{ ppm/year}$ ) and temperature ( $< 1 \text{ ppm/}^\circ\text{C}$ ).
- <sup>0</sup>Thermal hysteresis results showed some specimen-to-specimen variation. Although the lowest hysteresis results were for "as extruded" condition, it is suggested to test more specimens in each condition to have more conclusive results.
- <sup>0</sup>Stabilization heat treatments increased CTE of 11P **Invar 36** (from  $0.2 \text{ ppm/}^\circ\text{C}$  to approximately  $0.8 \text{ ppm/}^\circ\text{C}$ ) but it was not very clear that any particular heat treatment was more effective than any other for temporal stability. Almost all specimens were shrinking slower than  $1 \text{ ppm/year}$ .
- <sup>0</sup>Temperature change from  $38^\circ\text{C}$  to  $27.5^\circ\text{C}$  did not trigger any drastic new length drift rate except for "as extruded" condition.
- <sup>0</sup>Heat treatment H.T. #11 (Table 11) appears to be the best among the three-step heat treated **HP Invar 36** specimens with low thermal hysteresis, low thermal expansion and good temporal stability as well.
- <sup>0</sup>Simple two-step heat treatment H.T. #3 (Table II) did not result in any significant stability variation with temperature change and provided more confident, linear, temporal stability data at both temperatures. Additional CTE/thermal hysteresis testing is recommended.
- <sup>0</sup>I **HP Invar 36** in "as extruded" condition with 11 weeks or longer stabilization treatment at  $38^\circ\text{C}$  could have the lowest CTE, thermal hysteresis and good temporal stability at  $27.5^\circ\text{C}$ . This needs more investigation.

## 9. ACKNOWLEDGEMENTS

The investigation described in this paper was carried out by the Jet Propulsion Laboratory, California Institute of Technology, under contract with the National Aeronautics and Space Administration.

The authors would like to acknowledge the support of Mr. Steve Gunter, Technical Task Manager, ISS Cassini Subsystem. Also, the authors would like to thank Don Bass, U of A, Dimensional Stability Laboratory for performing most of the measurements described here.

## 10. REFERENCES

1. J. Marsh, "Alloys of Iron and Nickel," Vol I McGraw - Hill Book CO. 1938.
2. W. Shockley, *The Quantum Physics of Solid - I* Monograph B -1184, Dell Telephone Labs.
3. B.S. Lement, B.L. Averbach and M. Cohen, Trans. AMS 43, 1072-1097 (1951).
4. G. Z. Pupke, Phys. Chem. 207, 91 (1957).
5. W.S. McCain and R.E. Maringer, *DMIC Memorandum 207*, Battelle Mere, Inst. , Columbus, 011, 1965
6. C.W. Marshall, Proc. 21st. Natl. SAMPE Symp. Los Angeles, 1976

7. Physics and Application of Invar Alloys, Honda Memorial Series on Materials Science, No. 3, Maruzen Co Ltd, Tokyo, 1978, pp. 465-549
8. M.A. Hunter, *Low-Expansion Alloys*, Metals Handbook, American Society for Metals, Cleveland, 1948, p. 601
9. Iron - Nickel Alloys for Sealing Glasses and Ceramics, MIL-I-23011, U.S. Government Printing Office, March 24, 1974
10. *Metals Handbook*, 9th Edition, AMS, 1980 pp. 792-795
11. A.I. Zakharrow, B.V. Molotilow, L.V. Pastukhowa and N.A. Solov'yeva, *Fiz. Metal. Metalloved* 37, No. 2, 442-444 (1974)
12. T.P. O'Donnell and W.M. Rowe *Material Processing and Thermal Expansion Performance of Three Invar Alloys*, International Joint Conferences on Thermophysical Properties, 8th Thermal Expansion Symposium Proceeding, June 1981.
13. C.W. Marshall and R.E. Maringer, *Dimensions/ Instability*, Pergamon Press, New York 1977
14. A.D. White, *Applied Optics* 6, 1138 (1967)
15. T. Baer, F.V. Kowalski and J. I. Hall *Frequency Stabilization of a 0.633 Micron HeNe Longitudinal Zeeman Laser*, *Applied Optics* 19, 3173 (1980)
16. D. Hills and J.L. Hall, *Phys. Rev. Letters* 64, 1697-1700 (1990)
17. J.B. Berthold and S.F. Jacobs, *Ultraprecise Thermal Expansion Measurements of Several Low Expansion Material*, *Appl. Opt.* 15, (1976).
18. D.E. Schwab, S.T. Jacobs and S.F. Johnston *Isothermal Dimensional Instability of Invar*, 29th National SAMPE Symposium, pp. 169-184, April 3-5, (1984)
19. J. W. Berthold, S. F. Jacobs and M.A. Norton *Dimensional Stability of Fused Silica, Invar and Several Ultra-low Thermal Expansion Materials*, *Metrologia*, 13, 9-16 (1977)
20. S.F. Jacobs, *Dimensional Stability of Materials Useful in Optical Engineering*, *Optics Acts* 33, 1377-1388 (1986).
21. J.M. Steele, D.A. Thompson, S.F. Jacobs and D.L. Bass, *Temperature and Age Effects on Temporal Stability of Invar*, *SPIE Proc. V1752/10*, July 1992, San Diego, CA.
22. S.F. Jacobs *Variable Invariable - Dimensional Instability with Time and Temperature*, *SPIE, C.R.* July 1992, San Diego, CA.
23. W.M. Sokolowski, *Test Plan for Dimensional Stability Evaluation*, JPL Internal Report, March 1992.

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

Element	Metals Technology	Atlas Testing	Specialty Alloy	JPL	Desirable Composition
c	0.01	0.00s	0.002	0.01	<0.01
Mn	0.01	0.01	<0.001	<0.004	<0.02
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	<0.02
Se	0.0001		<0.00001		
Ni	36/24	36.0	36.0	36.8	36.0±0.1
Fe	REM	REM	REM	REM	REM

**Note:**

JPL results should be treated as the most reliable in light of the methods employed.

**TABLE II**  
**Dimensional Stability Test Specimens**

MATERIAL	ID	TEMPORAL STABILITY TEST	CTE/ HISTERESIS TEST	TOTAL
HP Invar 36:				
As Received	R	RI, R2	R3	3
H.T. #1	H1	Hi/1; Hi/2; Hi/3	H 1/4	4
H.T. #11	H11	H11/1; H11/2; H11/3	H11/4; H11/5; H11/6	6
H.T. #12	H12	H12/1; H12/2; H12/3	H12/4; H12/5	5
H.T. #2	H2	H2/1; H2/2; H2/3	H2/4; H2/5; H2/6	6
H.T. #3	H3	H3/1; H3/2; H3/3	H3/4; H3/5; H3/6	6
TOTAL NUMBER OF SPECIMENS		17	13	30

NOTES:

**HP Invar 36**

H.T. #1 "Annealing at 788°C/30 min., slow cool.  
 °Stress relief at 316°C/1 hr.  
 \*Aging at 93°C/48 hours

H.T. #11 Specimens were rough machined, annealed and final machined before stress relief and aging cycles per H.T. #1.

H.T. #12 H.T. #1 + 93°/28.5 days.

H.T. #2 °Annealing at 788°C/30min., slow COOL.  
 °Aging at 93°C/96 hrs.

H.T.#3 "Stress relief at 316°C/1 hr.  
 "Aging at 93°C/48 hrs.

TABLE 111

## CTE/Thermal Hysteresis Test Results

+50°C MATERIAL	ID	CTE	CTE	Hysteresis
		0°C to 25°C	-50°C to +50°C	-50°C to
		[ppm/°C]	[ppm/°C]	[ppm/Cycle]
<b>HP Invar</b>				
As Received	R3	0.20	0.29	0.28
H.T. #1	H1/4	0.71	<b>0.80</b>	1.05
	H11/5	0.76	0.85	0.12
	H11/6	0.76	0.82	0.63
H.T. #12	H12/4	0.77	0.82	<b>0.50</b>
	H12/5	0.74	<b>0.81</b>	2.70
H.T. #2	H2/6	0.70	0.81	2.70

TABLE IV

## TEMPORAL STABILITY TEST RESULTS

Material	ID	38° H. Winks		Ambient (27.5°C)	
		RATE (ppm/year)	Remarks	RATE (ppm/year)	6 Weeks Remarks
<b>HP Invar</b>					
As Received	R1	+2.4		0	
	R2	-2.7	Questionable	-1.2	Questionable
H.T. #1	H11/1	-5.6	Questionable	-0.1	Questionable
	H11/2	-2.2	Questionable	0	
	H11/3	-1.4	Questionable	-1.2	
H.T. #11	H11/1	-2.6	Questionable	-0.6	
	H11/2	-1.0		-0.6	
	H11/3	< 0	Noisy	-1.2	
H.T. #12	H12/1	-1.5		-0.4	Questionable
	H12/2	-0.6	Questionable	-0.6	Questionable
	H12/3	No Data		No Data	
H.T. #2	H2/4	-0.3		-1.2	Questionable
	H2/2	-1.0	Questionable	-0.1	
	H2/3	-0.8		-3.0	Questionable
H.T. #3	H3/1	-1.1		-1.2	
	H3/2	-0.8		0	
	H3/3	-0.8		-1.0	
Carbon/Carbon	C/C1	0		+1	
	C/C2	+1		+1	
	C/C3	-1		0	
SiC by CVD	SiC1	-0.5		-0.4	
	SiC2	-5	Questionable	-0.3	
SiC+Si	SiC/Si1	-2.5	Questionable	-0.1	
	SiC/Si2	-1.5	Questionable	0	
	SiC/Si3	-2.5		0	
F. Peek	Uni	-2.0		-2.0	
	45/45	-0.5		-6.7	Questionable
	0/90	+5.0		+3.2	
Ni/Cu Coated Gr/Epoxy	001	+30		+20	
	003	+30		+21	