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# Precise control of negative thermal expansion in stainless invar type alloy for astronomical telescopes

#### Hiromichi T. Fujii<sup>1</sup>, Naoki Sakaguchi<sup>1</sup>, Kotaro Ona<sup>1</sup> Yutaka Hayano<sup>2</sup>, Fumihiro Uraguchi<sup>2</sup>

<sup>1</sup> Shinhokoku Steel Corporation <sup>2</sup> National Astronomical Observatory of Japan



#### □ Thirty meter telescope (TMT)

- ✓ New class of extremely large telescope
- ✓ Observation of cosmic objects with unprecedented sensitivity

### Infrared imaging spectrograph (IRIS)

- Cooling down to cryogenic temperature to reduce the thermal noise
- Extremely low thermal expansion of the structural components is essential

#### □ Low thermal expansion alloys

- ✓ Invar alloy (Fe-36mass%Ni)
- CTE at cryogenic temperature is not low enough for infrared instruments



Thirty meter telescope (TMT) planned for construction in Hawaii (Courtesy of NAOJ)

#### Conventional invar alloy (Fe-36%Ni)

- ✓ Low thermal expansion around room temperature
- Not sufficient low CTE at cryogenic temperature as a structural material for IRIS

#### □ Stainless invar alloy (Fe-52%Co-10%Cr)

- Dimensional stability between cryogenic and room temperatures
- ✓ Adjustable CTE close to fused silica

#### □ Negative thermal expansion (NTE)

- ✓ Occurred between 200 and 340 K in stainless invar alloy
- ✓ Control of NTE could significantly reduce the dimensional difference to fused silica



Relative thermal expansion curves to a dimension at 100 K of the  $Fe_{66}Ni_{34}$  and the  $Fe_{37.8}Co_{51.8}Cr_{10.4}$  alloys. For comparison, a curve of the measured commercial fused silica is also shown.

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* Chikazumi, S.: J. Mag. Mag. Mater., 10, 113-119 (1979).
** Masumoto, H.: J. Jap. Inst. Met., 2, 141-146 (1938).
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Ternary phase diagram of Fe-Co-Cr alloy and the coefficients of thermal expansion at room temperature; Solid lines after Masumoto and dotted lines after Köster.

#### Microstructure

- ✓ Single face centered cubic (fcc:  $\gamma$ ) phase
- ✓ Ferromagnetic phase
- Optimum chemical composition
  - ✓ Co 52%, Fe 38%, Cr 10%
  - Minimum CTE region lies along the boundary between α and γ phases

#### Precise control of thermal expansion

 Accurate understanding of the effect of additional elements and microstructure on the thermal expansion characteristics

\* Masumoto, H.: J. Jap. Inst. Met., 2, 141-146 (1938).

- \*\* Köster, W.: Arch. Eisenhütt., 5, 17-23 (1932).
- \*\*\* Saito, H., Fujimori, H. and Saito, T.: Trans. JIM, 11, 68-71 (1970).



To obtain comprehensive knowledge on the negative thermal expansion characteristics in Fe-Co-Cr based alloys to develop a novel structural material for IRIS.

#### Research issues

- ✓ Effect of Ni addition on thermal expansion behavior
- ✓ Microstructural characterization of Fe-Co-Cr based alloys
- ✓ Effect of Cr element on negative thermal expansion at low temperature
- ✓ Origin of thermal expansion characteristics in Fe-Co-Cr based alloys

#### Sample preparation

- ✓ Material:  $Fe_{39}Co_{51-x}Cr_9Ni_x$  (x = 0, 1, 2)
- ✓ Methods: Induction melting, casting and/or forging
- ✓ Chemical analysis: Titration method
- Microstructural characterization
  - ✓ Optical microscopy
  - ✓ X-ray diffractometry
- Evaluation of material properties
  - $\checkmark$  Precise measurement of thermal expansion



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Casting

Measurement



# **Results and discussion**





Relative thermal expansion curves to a dimension at 100 K obtained from the annealed  $Fe_{39}Co_{51-x}Cr_{10}Ni_x$  (x = 0, 1, 2) specimens. For comparison, a relative thermal expansion curve of pure Fe is also shown by dotted line.

#### Drastic decrease of CTE

- Relative thermal expansion decreased with increasing Ni content
- ✓ CTE (100 ~ 200 K): 0.93 x 10<sup>-6</sup> /K
- ✓ CTE (200 ~ 300 K): -0.71 x 10<sup>-6</sup> /K

#### Effect of Ni addition

- One of the austenite (γ phase) stabilizing elements
- Increase the ferromagnetic γ phase with much lower CTE





Optical micrographs taken from the annealed  $Fe_{39}Co_{51-x}Cr_{10}Ni_x$  (x = 0, 1, 2) specimens. The dark and bright regions in the micrographs of the  $Fe_{39}Co_{51}Cr_{10}$  and the  $Fe_{39}Co_{50}Cr_{10}Ni$  specimens correspond to bcc  $\alpha$  and fcc  $\gamma$  phases, respectively.

#### Mixed microstructure

 Both bcc α and fcc γ phases were observed by the contrast of dark and bright regions

#### □ Single microstructure

 Many linear twin boundaries formed on fcc γ phase were observed

## **Crystallographic microstructure**



XRD profiles obtained from the annealed  $Fe_{39}Co_{51-x}Cr_{10}Ni_x$  (x = 0, 1, 2) specimens. Circles and squares indicate the diffraction peaks from fcc  $\gamma$  and bcc  $\alpha$  phases, respectively.

| Materials  | Area fraction [%]  |             |
|--|--------------------|-------------|
|  | bcc $\alpha$ phase | fcc γ phase |
| Fe <sub>39</sub> Co <sub>51</sub> Cr <sub>10</sub>                 | 71.4               | 28.6        |
| Fe <sub>39</sub> Co <sub>50</sub> Cr <sub>10</sub> Ni              | 57.1               | 42.9        |
| Fe <sub>39</sub> Co <sub>49</sub> Cr <sub>10</sub> Ni <sub>2</sub> | 0                  | 100         |

**G** Stabilization of fcc phase

- ✓ Ni addition of 2% eliminated bcc phase
- ✓ Full fcc phase microstructure is essential to obtain low thermal expansion alloy in Fe<sub>39</sub>Co<sub>51-x</sub>Cr<sub>10</sub>Ni<sub>x</sub> alloy



Relative thermal expansion curves to dimensions at 100 K and 150 K obtained from the annealed  $Fe_{39}Co_{49}Cr_{10}Ni_2$  and the  $Cr_{94}Fe_5Sn$  specimens, respectively. The curve of the  $Fe_{66}Ni_{34}$  is also shown for comparison.

#### Invar effect

- ✓ Thermal excitation from high spin to low spin states with temperature rise
- ✓ High spin state: Ferromagnetic and high volume
- Low spin state: Antiferromagnetic and low volume

## Effect of Cr on thermal expansion

- ✓ Antiferromagnetic element
- ✓ Acceleration of thermal excitation to low spin state between 200 and 340 K
  - $\rightarrow$  Negative thermal expansion

#### Natural thermal expansion

- ✓ Anharmonic effects of lattice oscillation (curve c)
- ✓ All atoms are kept as only high spin state

#### Invar effect

- Thermal excitation from high spin to low spin states with contraction
- ✓ Cancellation of natural thermal expansion by magnetic contraction (curve b)
- Thermal expansion in stainless invar alloy
  - Negative thermal expansion caused by acceleration of thermal excitation from high spin to low spin states (curve a)



Potential energy curves predicted from Morse's equation in Fe atoms of high spin and low spin states. Inter-atomic distances at some temperatures in the stainless invar type alloy, the invar type alloy and the natural thermal expansion are shown by circles.





A relative thermal expansion curve obtained from the  $Fe_{38.4}Co_{49.7}Cr_{10.1}Ni_{1.8}$  alloy with precisely controlled CTE between 100 K and room temperature. For comparison, the curve obtained from the commercial fused silica is also shown.

#### Control of thermal expansion

- Precise adjustment of chemical composition based on the experimental results
- Dimensional change between 100 K and room temperature of a stainless invar alloy was matched with that of a fused silica
- ✓ Average CTE between 100 K and room temperature was approximately 0.13 x 10<sup>-6</sup> /K



- The average CTE between cryogenic and room temperatures drastically decreased by the addition of 2% Ni in the Fe<sub>39</sub>Co<sub>51-x</sub>Cr<sub>10</sub>Ni<sub>x</sub> alloy.
- 2. Antiferromagnetic Cr elements included in the Ni-added stainless invar alloys would accelerate thermal excitation of the electrons from ferromagnetic high spin state to antiferromagnetic low spin state.
- 3. The desired dimensional change including zero between cryogenic and room temperatures can be obtained by precise control of negative thermal expansion in the Ni-added stainless invar alloys.

Observation accuracy of infrared instrument mounted on astronomical telescope could be drastically improved by the structural components made by the Ni-added stainless invar alloys.

Thank you for your kind attention.



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> Hiromichi T. Fujii +81-49-242-1955 fujii@shst.co.jp www.shst.co.jp �



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- 者: Hiromichi T. Fujii, Naoki Sakaguchi, Kotaro Ona, Yutaka Hayano, Fumihiro Uraguchi
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#### 概 要:

Thermal expansion characteristics of Fe<sub>39</sub>Co<sub>51</sub>Cr<sub>10</sub> based stainless invar alloys were investigated with the aim of developing a novel structural material used at cryogenic temperature for infrared instruments mounted on astronomical telescopes. Ni-added stainless invar type alloys Fe<sub>39</sub>Co<sub>49</sub>Cr<sub>10</sub>Ni<sub>2</sub> were found to exhibit anomalous thermal expansion behavior in the low temperature range between 100 K and room temperature. According to the measured thermal expansion curves, these alloys expanded with increasing temperature from approximately 100 K to 200 K and contracted from 200 K to 300 K. The coefficients of thermal expansion in each temperature range were estimated to be approximately 0.93 x 10<sup>-6</sup> /K and -0.71 x 10<sup>-6</sup> /K, respectively. As the results of thermal expansion measurements of Cr based alloys, similar negative thermal expansion behavior was observed in the same temperature range between 200 K and 300 K. These findings suggest that the Cr element plays a key role in the negative thermal expansion in the  $Fe_{39}Co_{49}Cr_{10}Ni_2$  alloys. We concluded that the dimensional change between 100 K and room temperature could be precisely controlled in the extremely low thermal expansion range by controlling the negative thermal expansion. The coefficient of thermal expansion between 100 K and 300 K was achieved to be approximately 0.13 x 10<sup>-6</sup> /K in the Fe<sub>384</sub>Co<sub>497</sub>Cr<sub>101</sub>Ni<sub>18</sub> alloys developed in this study. As this value was close to that of the fused silica used for infrared instruments, the developed alloys can be a powerful tool for high precision observation in astronomical telescopes.

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