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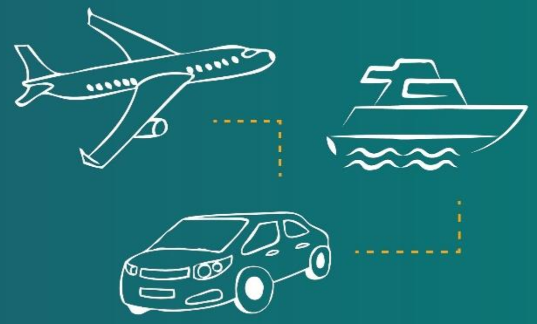

COMBINED STRENGTH. UNSURPASSED INNOVATION

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Novel tooling materials with extremely high dimensional stability for press forming of CFRTP

Hiromichi T. Fujii*, Naoki Sakaguchi, Haruyasu Ohno, Kotaro Ona

* fujii@shst.co.jp

Principal researcher

SHINHOKOKU STEEL CORP.

(SHINHOKOKU MATERIAL CORP., 10/1/2021 ~)

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Personal introduction

- Speaker Name: Hiromichi T. Fujii
- Company: Shinhokoku Steel Corp.
  (Shinhokoku Material Corp., 10/1/2021 ~)
- Job Title: Principal researcher
- Brief bio information;
  - Visiting Scholar, University of Cambridge, 2008
  - Ph.D. (Engineering), Graduate School of Engineering, Tohoku University, 2009
  - Assistant Professor, Tohoku University, 2009-2018
  - Visiting Scholar, The Ohio State University, 2010-2011
  - Principal Researcher, Shinhokoku Steel Corp., 2019-

Invar alloy used as a tooling material of CFRP molding

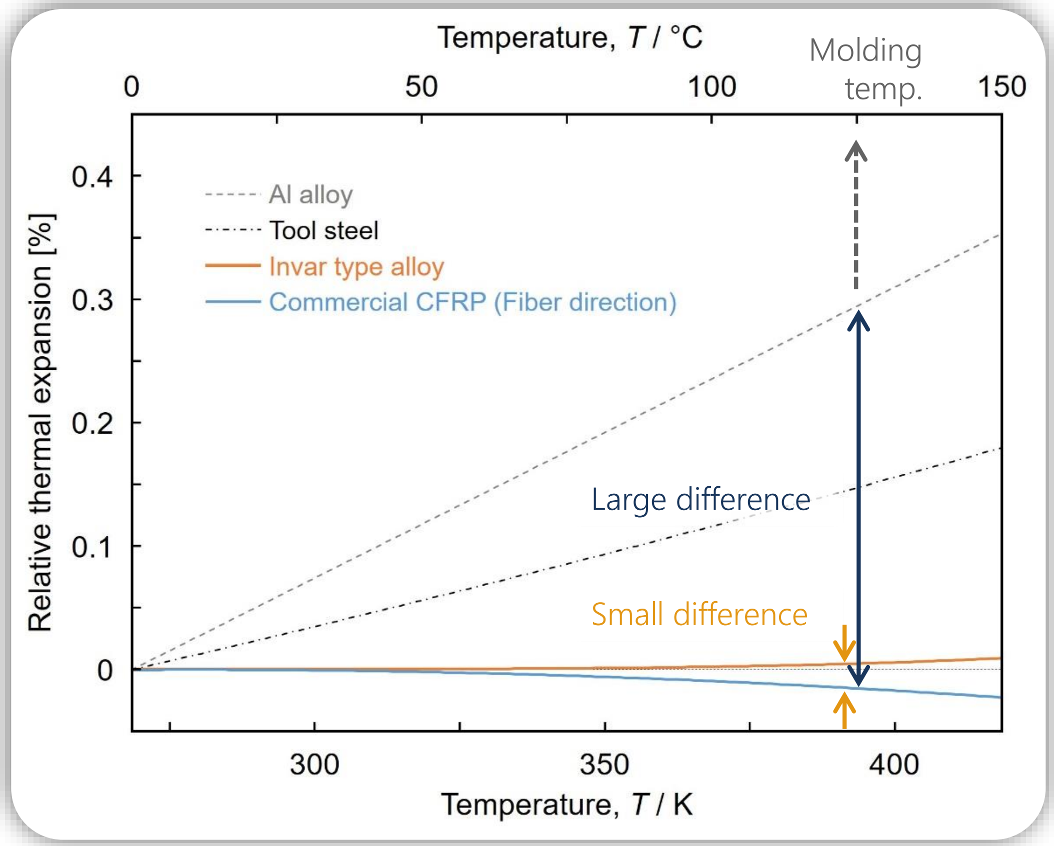
Invar alloy

- ✓ Fe-36mass%Ni alloy
- ✓ Low thermal expansion (R.T. ~ 200ºC)
- ✓ Tooling material for CFRP components in aerospace industry

Requirements for tooling material

- ✓ Close dimensional change to CFRP between room and molding temps.
  (Match the CTE* values of production and tooling materials)
- ✓ High durability
- ✓ Thermal stability

* Coefficient of thermal expansion



Comparison of relative thermal expansion curves to a dimension at room temperature between tooling materials.

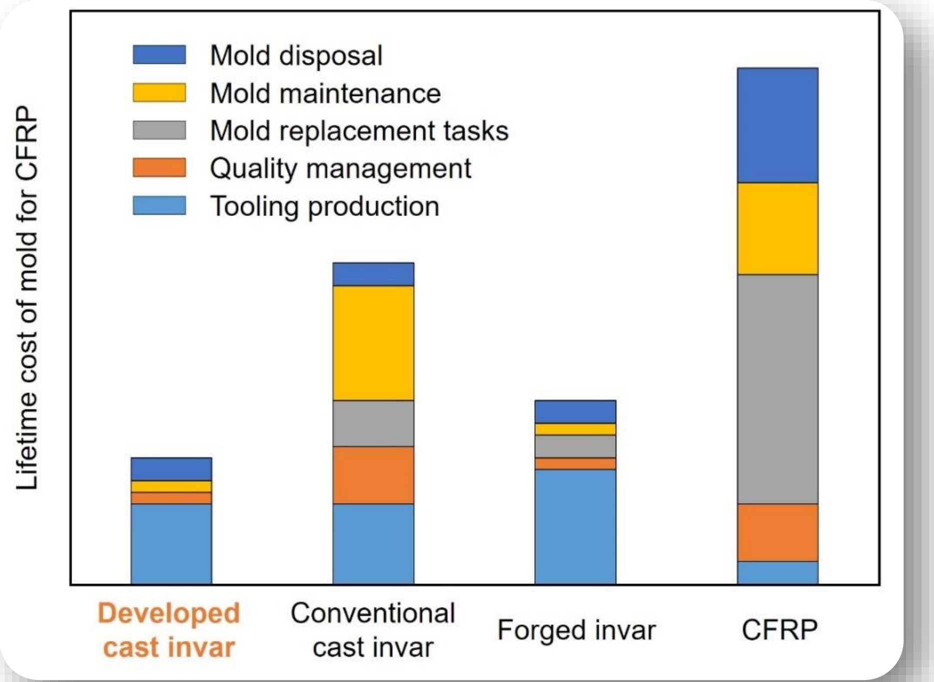
Background and research motivation

CFRTP components

- ✓ Weight reduction
- ✓ Mass production
- ✓ High dimensional accuracy

Potential of tooling technology

- ✓ Precise molding
- ✓ Intricate structure with small curvature
- ✓ Stable production of high quality components
- ✓ Cost reduction

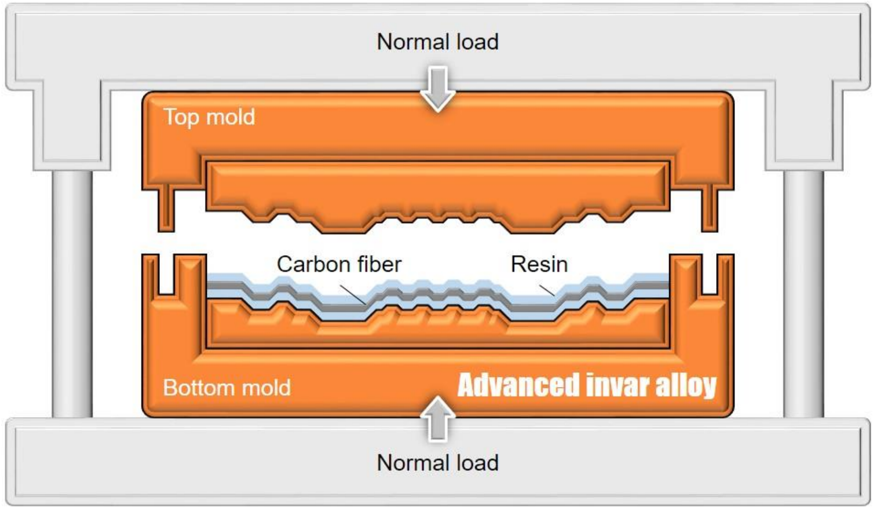


Summary of lifetime cost in each tooling material for small CFRP component with an intricate structure.

Press forming with advanced invar alloy molds

Tooling materials for press forming

- ✓ Near-net shape
  (As-cast structure)
- ✓ High durability
  (Decrease in aged deterioration)
- ✓ Thermal stability
  (Precise molding of intricate structure)
- ✓ Maintenance free
  (Automation of factory, mass production)



Press forming with the advanced invar alloy molds that allow mass production of the small CFRP components with intricate structure.

Cast invar alloys with supreme durability can be a powerful tool for CFRTP press forming

Objective

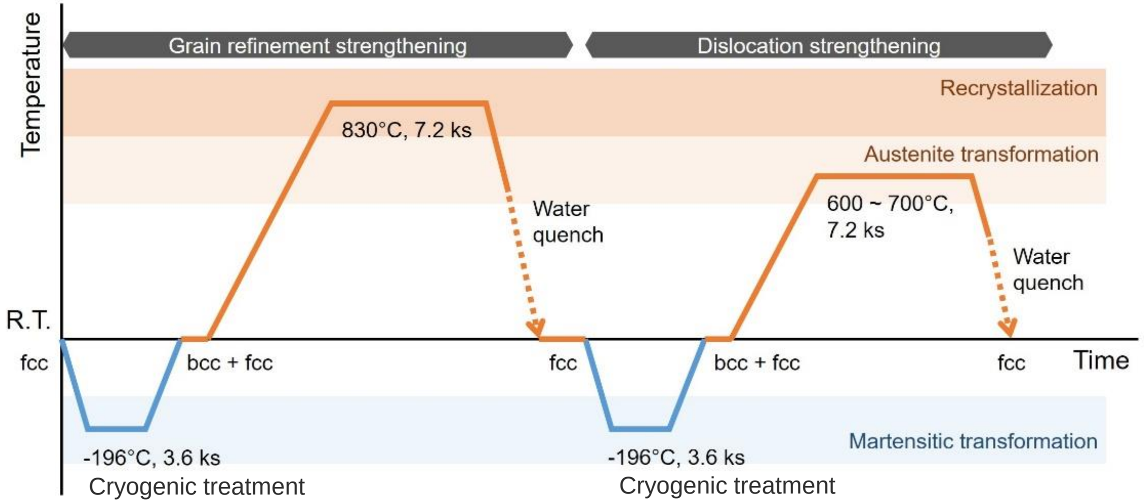
To reveal the relationship between chemical composition and thermal expansion characteristics in the low thermal expansion alloys, and the effects of microstructural control on mechanical properties.

Sample preparation

Chemical composition

✓ $Fe_{64-x-y}Ni_{31+x}Co_{5+y}$ alloys
(Base composition: $Fe_{65}Ni_{35}$, $Fe_{64}Ni_{31}Co_{5}$ and $Fe_{55}Ni_{28}Co_{17}$)

Thermal treatment



Thermal treatment developed for strength improvement

Developed cast invar alloy

Casting → **Developed thermal treatment** → Finish processing

Near-net shape | High rigidity, Low CTE

Fine grains

10mm

Conventional cast invar alloy

Casting → Thermal treatment → Finish processing

Near-net shape | Low CTE

Coarse grains

10mm

Forged invar alloy

Casting → Forging → Precision machining → Thermal treatment → Finish processing

Ingot | High rigidity | Low CTE

Fine grains

10mm

Results and discussion

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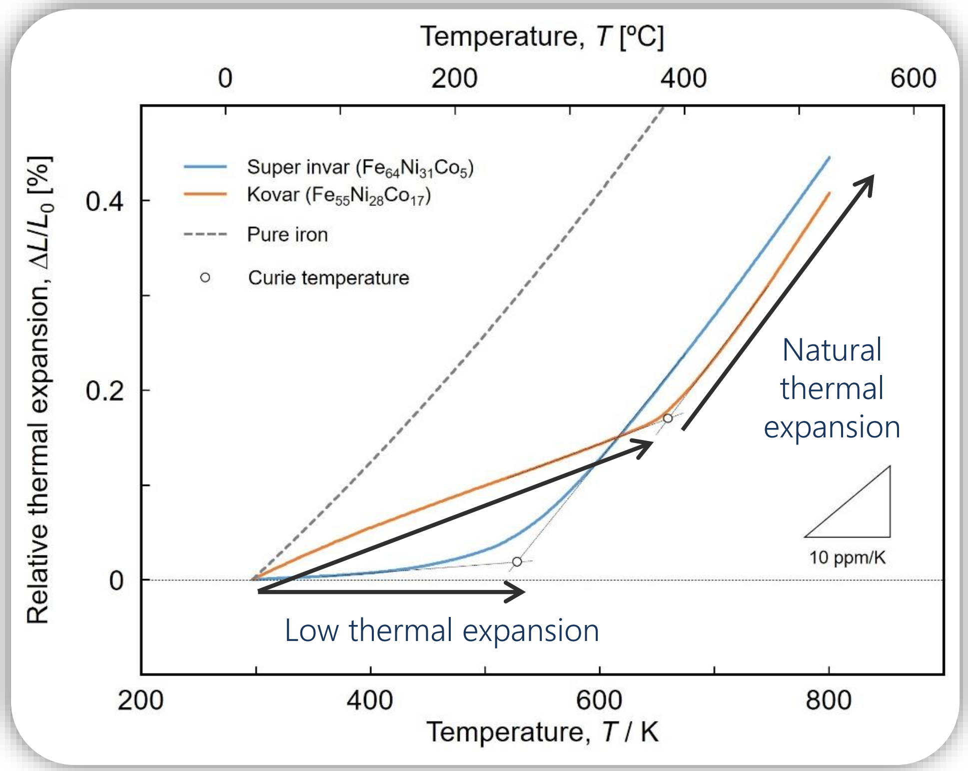
Conventional low thermal expansion alloys

- **Low thermal expansion characteristics**
  - ✓ Magnetic contraction and natural thermal expansion are canceled by each other below Curie temperature
  - ✓ Temperature range can be expanded by increasing Curie temperature
- **Curie temperature of iron based alloys**
  - ✓ Strongly depend on their chemical compositions



Thermal expansion curves obtained from super invar ($Fe_{64}Ni_{31}Co_5$) and kovar ($Fe_{55}Ni_{28}Co_{17}$) alloys. For comparison, a curve of pure iron is also shown by dotted line.

Formulation of Curie temperature in low thermal expansion alloys

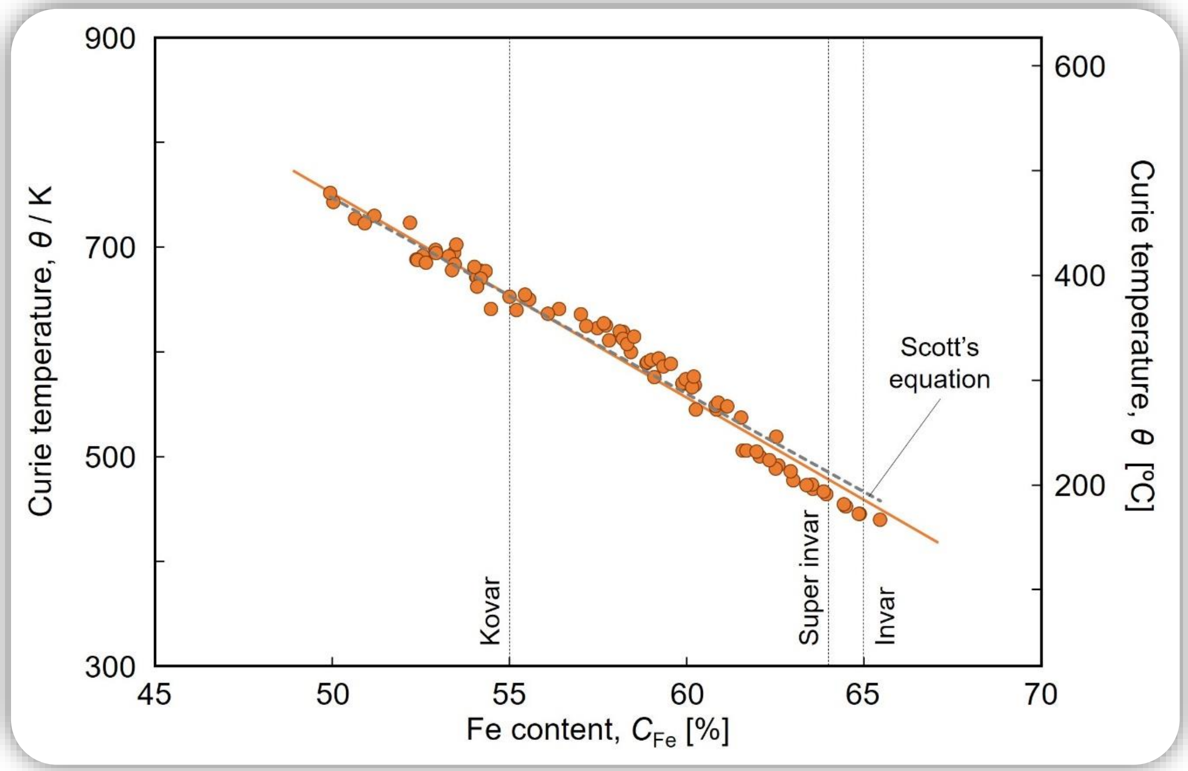
Dependence of Curie temperature on chemical composition

- ✓ Linear relationship between Fe content and Curie temperature
- ✓ Curie temperature could be raised by over 100°C compared to conventional alloys

Empirical equation of Curie temperature

- ✓ Scott's equation*

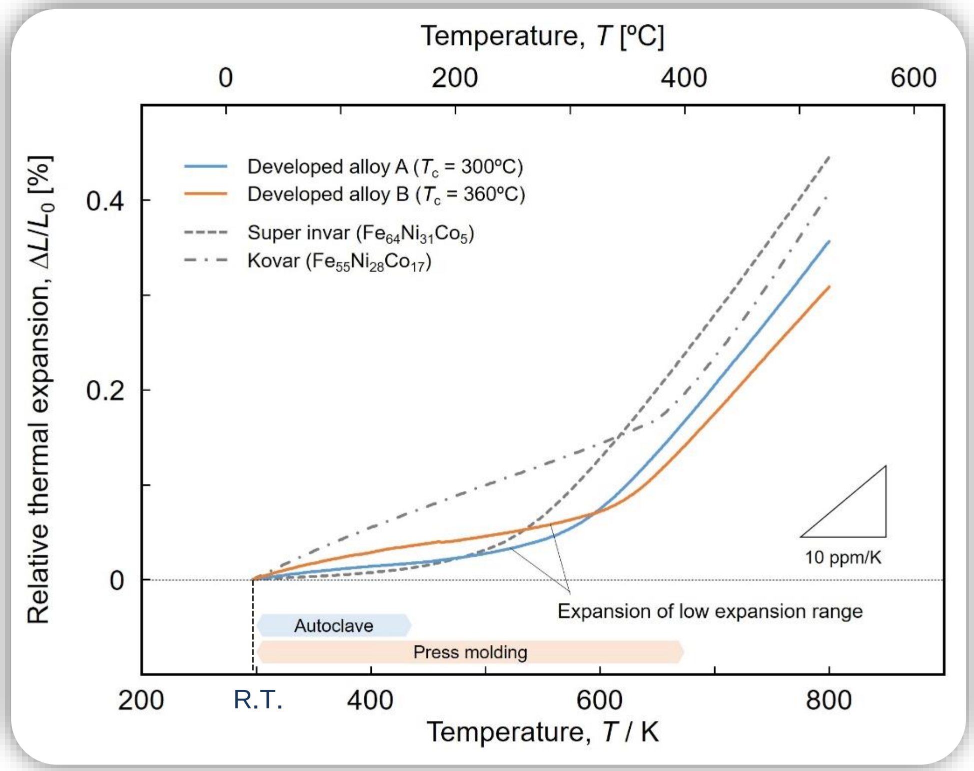
$$\theta = 19.5(C_{Ni} + C_{Co}) - 22C_{Mn} - 465$$
$$= 19.5(100 - C_{Fe}) - 465$$
$$= -19.5C_{Fe} + 1485$$



Relationship between Curie temperature and Fe content in the $Fe_{64-x-y}Ni_{31+x}Co_{5+y}$. Curie temperatures were estimated from inflection points of thermal expansion curves.

* "Physics and applications of invar alloys", *Maruzen Company*, (1978), 528-531.

Development of novel low thermal expansion alloys

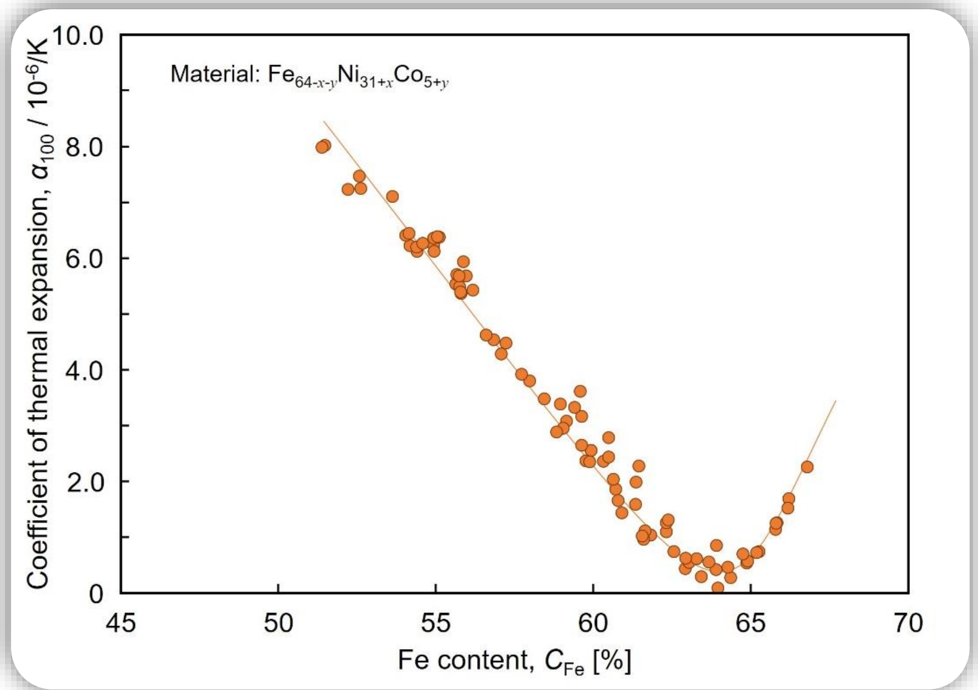


Thermal expansion curves obtained from newly developed alloys by controlling Curie temperature. For comparison, the curves of conventional super invar and kovar alloys are also shown by dotted lines.

- Control of low thermal expansion characteristics
  - ✓ Lower thermal expansion than conventional alloys
  - ✓ Expanded temperature range by increasing Curie temperature

Material	Curie temperature [ºC]	Average CTE between room and specific temperatures [10^{-6} / ºC]		
		R.T. ~ 200ºC	R.T. ~ 300ºC	R.T. ~ 400ºC
Developed alloy A	314	1.3	1.9	4.4
Developed alloy B	363	2.3	2.3	3.7
Super invar	250	1.7	4.1	6.9
Kovar	378	5.2	4.9	5.4

Control of coefficient of thermal expansion



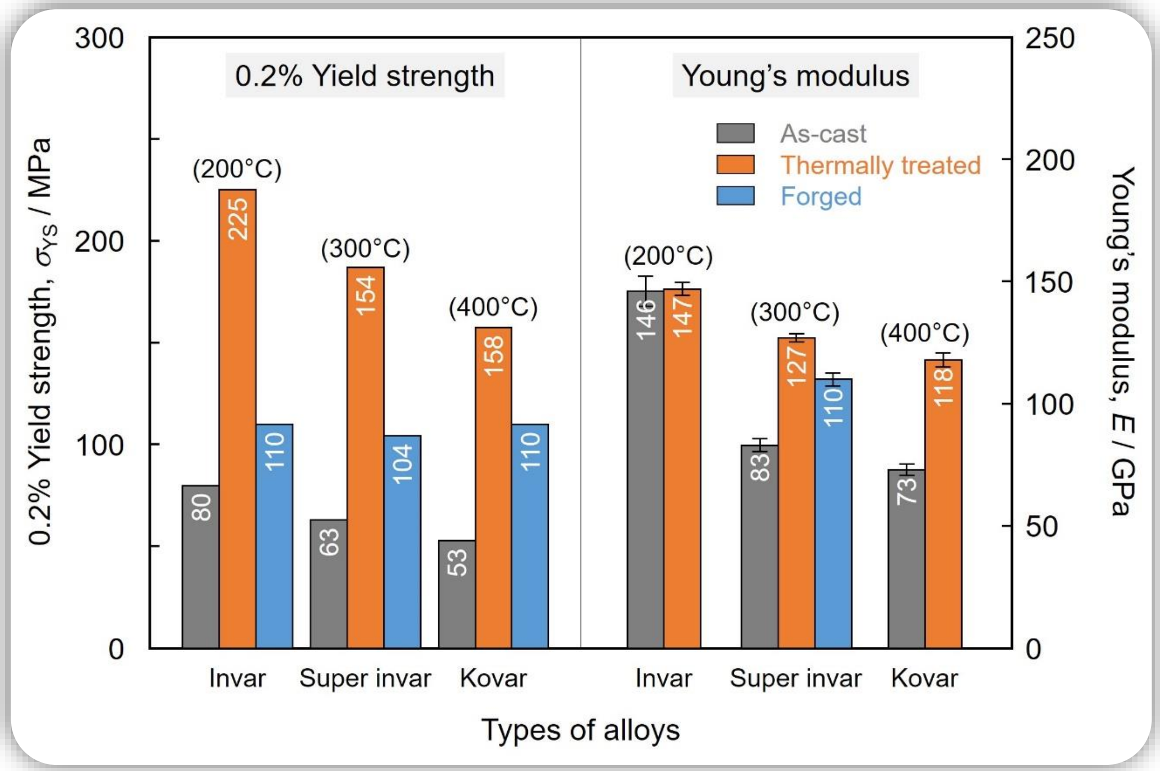
Relationship between average coefficients of thermal expansion and Fe content. The average coefficients of thermal expansion were measured between room temperature and 100°C.

- Average coefficient of thermal expansion
  - ✓ Optimum Fe content for minimum CTE could be easily determined from the concave relationship
  - ✓ Controllable by adjusting only Fe content with 0.1% accuracy in desired temperature range

Mechanical properties improved by heat treatment

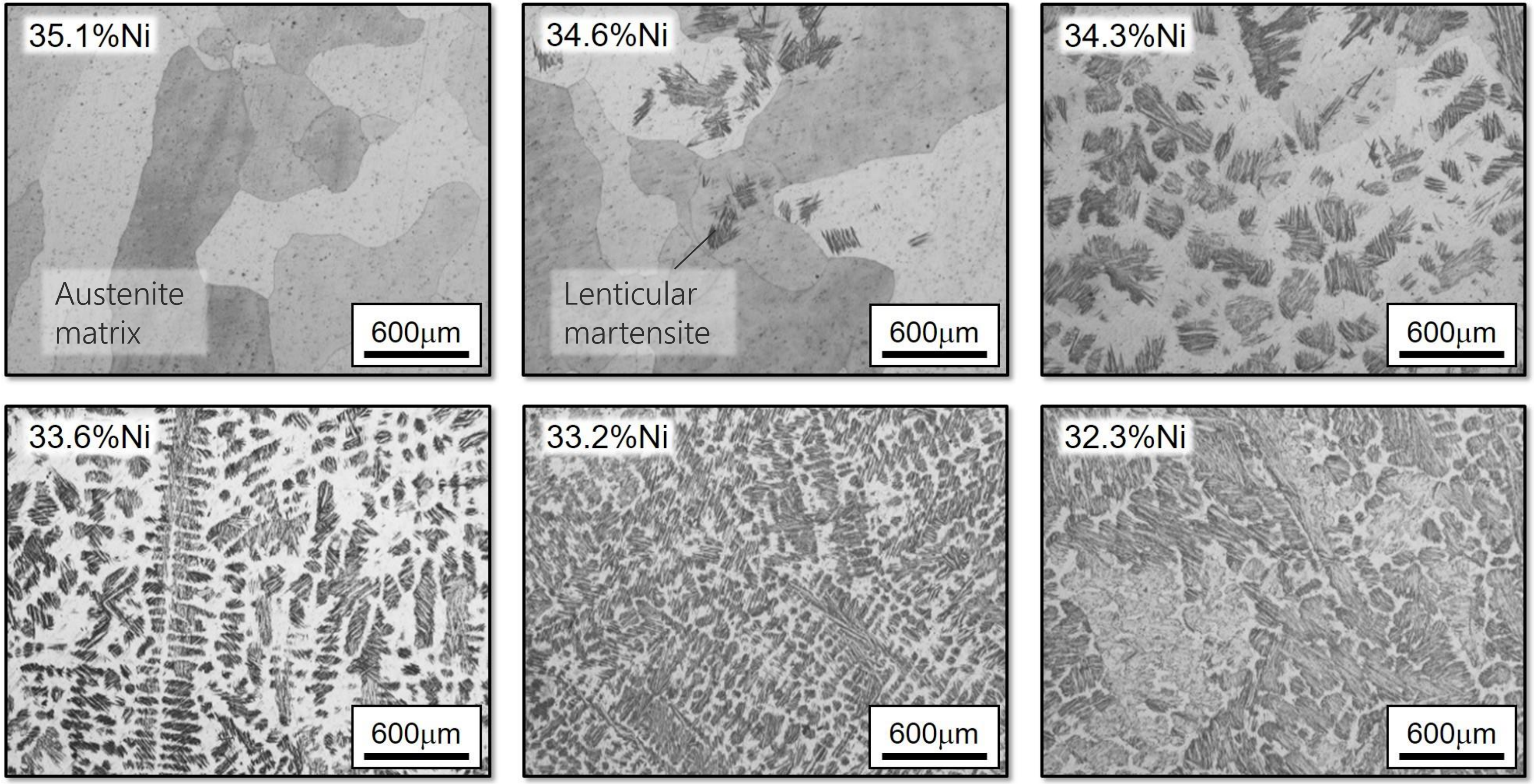
Effect of thermal treatment on mechanical properties

- ✓ Reverse austenite transformation from martensitic phase formed by cryogenic treatment
- ✓ Grain refinement and increase in dislocation density
- ✓ Drastic improvement of yield strength

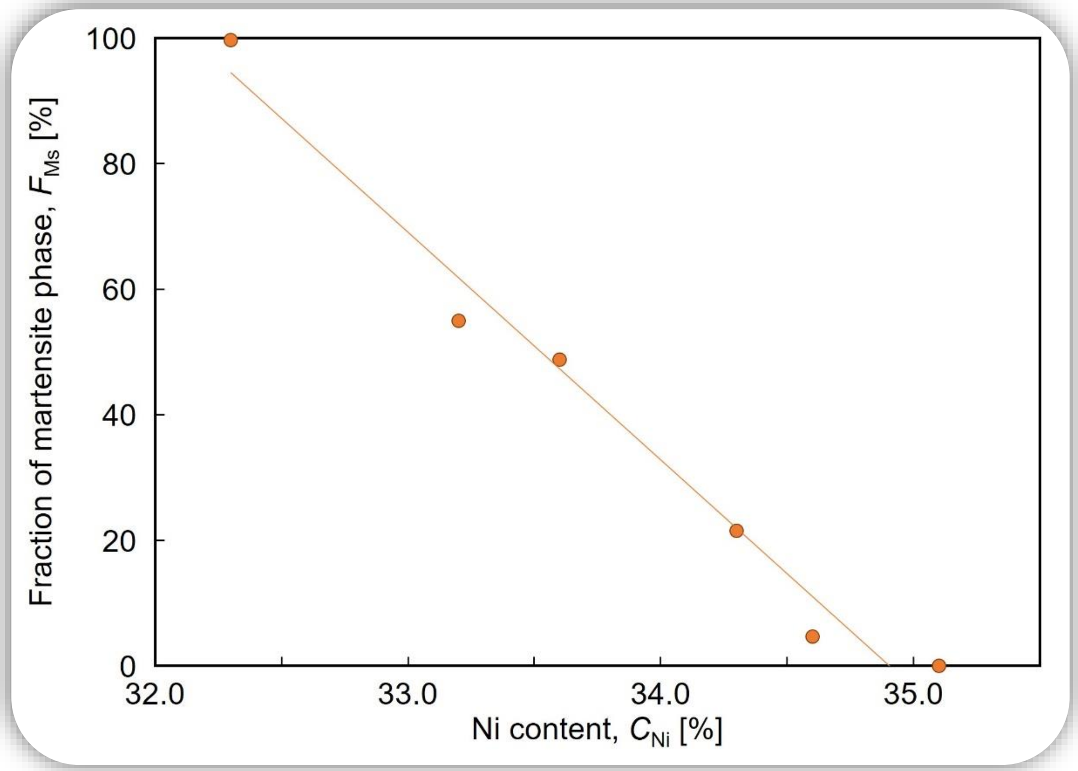


0.2% yield strength and Young's modulus of the as-cast and the thermally treated invar, super invar and kovar alloys. For comparison, those of the forged alloys were also shown.

Formation of martensitic grains by cryogenic treatment



Microstructural control to improve mechanical properties

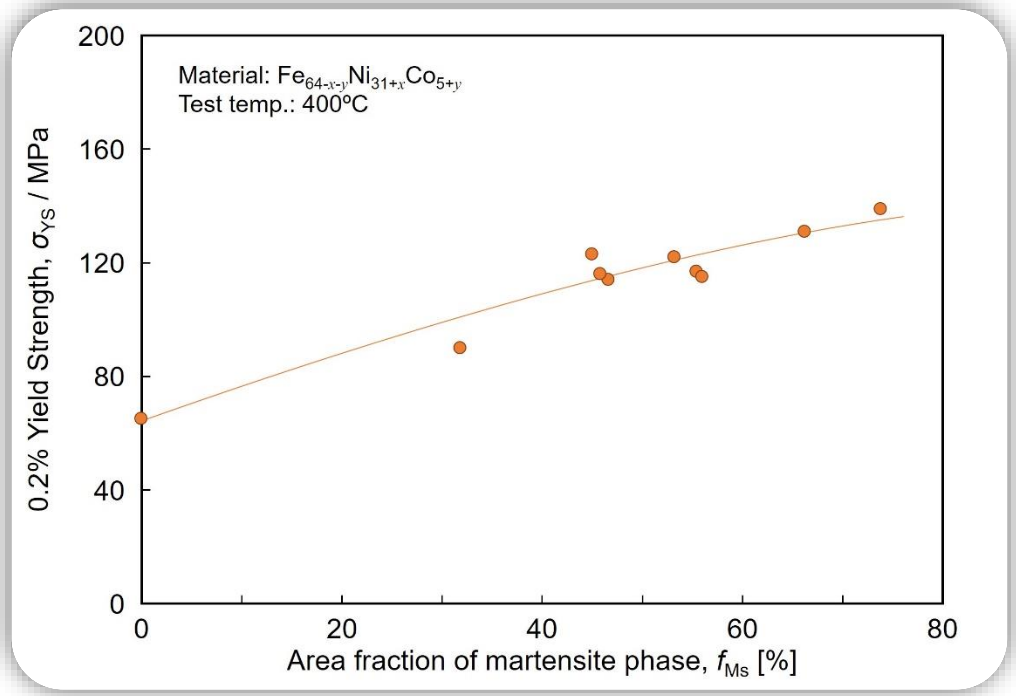


Relationship between area fraction of martensite phases formed during cryogenic treatment and Ni contents in the Fe-Ni based low thermal expansion alloys.

- **Control of martensitic transformation**
  - ✓ Linear relationship between area fraction of martensitic phase and Ni content
  - ✓ Drastic decrease with the slightest increase in Ni content

- **Cryogenic and heat treatments**
  - ✓ Fine and equiaxed grains microstructure
  - ✓ Mechanical properties could be improved by adjustment of only Ni content

Effect of martensitic phase on mechanical properties



Relationship between 0.2% yield strength after austenite transformation and the area fraction of martensite phases formed during cryogenic treatment by liquid nitrogen in the $Fe_{64-x-y}Ni_{31+x}Co_{5+y}$ alloys.

- Mechanical properties determined by area fraction of martensitic phase
  - ✓ Yield strength and tensile strength increased with increasing the area fraction before plateauing at 60%.
  - ✓ There was no significant dependence of Young's modulus on the area fraction

Summary

- ✓ The Curie temperatures of the developed materials were linearly decreased with increasing Fe content. They could be raised above 350ºC without loss of their low thermal expansion characteristics by the adjustment of only Fe content.

- ✓ The average CTE in the arbitrary temperature range could be controlled by the adjustment of Fe content. Improvement of mechanical properties of the developed materials through reverse martensitic transformation was dependent with the area fraction of martensite phases formed during cryogenic treatment.

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SHINHOKOKU STEEL CORPORATION

Hiromichi T. Fujii, Ph.D.
fujii@shst.co.jp
Principal researcher
SHINHOKOKU STEEL CORP.

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概　要；

Press forming of carbon fiber reinforced thermoplastic (CFRTP) pre-preg has been attracted attention as a promising technique to drastically reduce its production cycle time. Consideration of thermal expansion differences between tooling material and CFRTP part is essential to achieve the desired dimensional tolerances in the press forming. An invar alloy is known as one of low thermal expansion materials and has been used as a tooling material for the autoclave molding. However, its low thermal expansion characteristics is lost above approximately 200ºC and normal thermal expansion occurs above the Curie temperature of 240ºC. In the press forming of CFRTP generally conducted above 250ºC, therefore, the invar alloy cannot be used as a tooling material to achieve precise forming. In this study, novel tooling materials with high dimensional stability between room and press forming temperatures were developed with the aim of precise press forming of CFRTP components.

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