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(54) **MEDIUM-ENTROPY ALLOY HAVING EXCELLENT CRYOGENIC CHARACTERISTICS**

LEGIERUNG MIT MITTELHOHER ENTROPIE UND HERVORRAGENDEN KRYOGENEN EIGENSCHAFTEN

ALLIAGE À ENTROPIE MOYENNE AYANT D'EXCELLENTE CARACTÉRISTIQUES CRYOGÉNIQUES

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- **HAKARU MASUMOTO ET AL: "The Strain Gage Factor and Electrical Properties of Fe-Cr-Co-Ni Alloys", JOURNAL OF THE JAPAN INSTITUTE OF METALS AND MATERIALS, vol. 47, no. 10, 31 October 1983 (1983-10-31), pages 879-884, XP055679916,**
- **Y. H. JO ET AL: "Cryogenic strength improvement by utilizing room-temperature deformation twinning in a partially recrystallized VCrMnFeCoNi high-entropy alloy", NATURE COMMUNICATIONS, vol. 8, no. 1, 12 June 2017 (2017-06-12), XP055680269, DOI: 10.1038/ncomms15719**
- **GALI A. et al.: "Tensile Properties of High- and Medium-entropy Alloys", Intermetallics, vol. 39, 2013, pages 74-78, XP028591065,**

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**EP 3 660 178 B1**

- **GLUDOVATZ et al.: "Exceptional Damage-tolerance of a Medium-entropy Alloy CrC'oNi at Cryogenic Temperatures", Nature Communications, 2 February 2016 (2016-02-02), XP055563185,**

**Description****Technical Field**

5 **[0001]** The present invention relates to a medium-entropy alloy (MEA) having excellent cryogenic mechanical properties, in which inexpensive Fe is included in an amount of 55 to 62.5 at% to thus exhibit high price competitiveness, and moreover, in which face-centered cubic (FCC) and body-centered cubic (BCC) phase stability may be adjusted through control of alloying elements to thus cause deformation-induced phase transformation during cryogenic deformation, thereby realizing excellent cryogenic mechanical properties.

**Background Art**

10 **[0002]** A high-entropy alloy (HEA) is a multielement alloy obtained by alloying five or more constituent elements at a similar ratio without a major element of the alloy. A high-entropy alloy is a metal material having a single-phase structure, such as a face-centered cubic (FCC) phase or a body-centered cubic (BCC) phase, without forming intermetallic compounds or intermediate phases, due to high entropy of mixing in the alloy.

15 **[0003]** In particular, a Co-Cr-Fe-Mn-Ni-based high-entropy alloy has excellent cryogenic properties, high fracture toughness and high corrosion resistance, and is thus receiving attention as a material suitable for use in extreme environments.

20 **[0004]** Two important factors in designing such a high-entropy alloy are the composition ratio of the constituent elements of the alloy and the configurational entropy of the alloy system.

25 **[0005]** Here, the composition ratio of the high-entropy alloy is discussed first. A high-entropy alloy has to be composed of at least five elements, and the fraction of each of the constituent elements of the alloy is set to the range of 5 to 35 at%. Furthermore, when another element is added in the production of a high-entropy alloy, in addition to the main alloying elements, the amount thereof should be 5 at% or less.

30 **[0006]** Also, alloys are typically divided into high-entropy alloys, medium-entropy alloys (MEAs), and low-entropy alloys (LEAs), depending on the configurational entropy ( $\Delta S_{conf}$ ) of the composition of alloying elements, and are classified according to the conditions of Equation 2 below based on the configurational entropy value determined by Equation 1 below.

[Equation 1]

$$35 \quad \Delta S_{conf} = -R \sum_{i=1}^n X_i \ln X_i$$

40 (R: Gas constant,  $X_i$ : mole fraction of i element, n: number of constituent elements)

[Equation 2]

$$45 \quad \begin{aligned} \Delta S_{conf} &\leq 1.0 \cdot R \text{ (LEAs),} \\ 1.0 \cdot R &\leq \Delta S_{conf} \leq 1.5 \cdot R \text{ (MEAs),} \\ 50 \quad 1.5 \cdot R &\leq \Delta S_{conf} \text{ (HEAs)} \end{aligned}$$

55 **[0007]** For a  $\text{Co}_{20}\text{Cr}_{20}\text{Fe}_{20}\text{Mn}_{20}\text{Ni}_{20}$  (at%) alloy, which is a representative cryogenic FCC-based high-entropy alloy, the alloying elements that are added are expensive, resulting in low price competitiveness. Accordingly, despite the excellent cryogenic properties thereof, the above alloy makes it difficult to serve as a replacement for existing steel materials in marine plants, LNG container materials, cryogenic tanks, and ship/marine materials.

**[0008]** Therefore, the industrialization of high-entropy alloys is essentially required to ensure price competitiveness

through control of alloying elements and also to realize excellent cryogenic properties.

[Citation List]

- 5 [0009] (Patent Document) U.S. Patent Application Publication No. 2002/0159914  
 [0010] (Non-Patent Document) 1. B. Gludovatz, et al., "A fracture-resistant high-entropy alloy for cryogenic applications", Science, 345 (2014) 1153-1158.  
 [0011] H. Masumoto et al., JOURNAL OF THE JAPAN INSTITUTE OF METALS AND MATERIALS, vol. 47, no. 10, pages 879-884 refers to measurements of the strain gage factor and electric properties of Fe-Cr-Co-Ni alloys. WO  
 10 90/00629 A1 discloses Fe-Mn-Al-C based alloys for suppressing vibration noise. US 3673010 A deals about cold-workable permanent magnet alloys containing Co, Ni, Cr and Fe.

## Disclosure

### 15 Technical Problem

[0012] Accordingly, an objective of the present invention is to provide a medium-entropy alloy, which is capable of exhibiting superior mechanical properties by causing deformation-induced phase transformation at cryogenic temperatures, as well as ensuring price competitiveness by developing an alloy including reduced amounts of expensive alloying  
 20 elements, in lieu of a conventional Co-Cr-Fe-Mn-Ni-based alloy.

### Technical Solution

[0013] The invention is defined by the appended claims. Any disclosure lying outside the scope of said claims is intended for illustrative as well as comparative purposes.  
 25

### Advantageous Effects

[0014] According to the present invention, a medium-entropy alloy is configured such that the amount of Fe, which is an inexpensive alloying element, is increased to the range of 55 to 62.5 at%, thus reducing the amounts of expensive  
 30 Co, Cr, and Ni elements that are added, thereby ensuring price competitiveness, and moreover, the medium-entropy alloy has superior properties including tensile strength of 1024 MPa or more and elongation of 47% or more at a cryogenic temperature (77 K). Moreover, according to an embodiment of the present invention, the medium-entropy alloy includes a metastable FCC phase at room temperature (298 K) and causes deformation-induced phase transformation from the metastable FCC phase into a BCC phase upon deformation at a cryogenic temperature to thus exhibit strengthening  
 35 effects, ultimately obtaining further improved cryogenic mechanical properties.

### Description of Drawings

#### 40 [0015]

FIG. 1 shows the results of measurement of X-ray diffraction (XRD) of Co-Cr-Fe-Ni-based medium-entropy alloys of Comparative Examples 1 and 2 and Examples 1 to 4 according to the present invention;  
 FIG. 2 shows the results of tensile testing at room temperature (298 K) of the Co-Cr-Fe-Ni-based medium-entropy  
 45 alloys of Comparative Examples 1 and 2 and Examples 1 to 4 according to the present invention;  
 FIG. 3 shows the results of tensile testing at a cryogenic temperature (77 K) of the Co-Cr-Fe-Ni-based medium-entropy alloys of Comparative Examples 1 and 2 and Examples 1 to 4 according to the present invention; and  
 FIG. 4 shows the analytical results of electron backscatter diffraction (EBSD) for phase transformation upon deformation at room temperature and at a cryogenic temperature of the Co-Cr-Fe-Ni-based medium-entropy alloy of  
 50 Example 3 according to the present invention.

### Best Mode

[0016] Hereinafter, a detailed description will be given below of preferred embodiments of the present invention with reference to the appended drawings, but the present invention is not limited to the following examples.  
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[0017] The present inventors have performed thorough study in order to obtain excellent mechanical properties in cryogenic environments while increasing price competitiveness of high-entropy alloys having superior mechanical properties in cryogenic environments, and thus have ascertained that the amount of Fe, which is an inexpensive element,

is remarkably increased to the range of 55 to 62.5 at% compared to conventional high-entropy alloys, and the amounts of alloying elements other than Fe are adjusted, whereby deformation-induced phase transformation may occur during deformation due to changes in FCC and BCC phase stability, resulting in excellent cryogenic mechanical properties.

5 [0018] In particular, the present inventors have revealed that when an alloy is designed so as to include an FCC phase in a metastable state at room temperature, deformation-induced phase transformation from the FCC phase in a metastable state into a BCC phase may occur during deformation in a cryogenic environment, thereby further improving cryogenic mechanical properties, which culminates in the present invention.

10 [0019] In the present invention, when the phase in a metastable state is transformed into a phase in a stable state at the corresponding temperature through deformation-induced phase transformation during plastic deformation, it is judged to be a metastable phase. All of these phases are defined as a metastable phase.

[0020] If the amount of chromium (Cr) is less than 6 at%, the FCC phase is stabilized. On the other hand, if the amount thereof exceeds 15 at%, the BCC phase is stabilized. Hence, the amount of Cr falls in the range of 6 to 12.5 at%. Also, since the formation of the metastable FCC phase is more favorable in terms of improving cryogenic mechanical properties, the amount of chromium (Cr) more preferably falls in the range of 7.5 to 12.5 at%.

15 [0021] If the amount of iron (Fe) is less than 50 at%, the FCC phase is stabilized. On the other hand, if the amount thereof exceeds 64 at%, the BCC phase is stabilized. Since the formation of the metastable FCC phase is more favorable in terms of improving cryogenic mechanical properties, the amount of iron (Fe) in the invention falls in the range of 55 to 62.5 at%.

[0022] If the amount of cobalt (Co) is less than 13 at%, the FCC phase is stabilized. On the other hand, if the amount thereof exceeds 25 at%, the BCC phase is stabilized. Hence, the amount of Co falls in the range of 13 to 25 at%.

20 [0023] If the amount of nickel (Ni) is less than 13 at%, the BCC phase is stabilized. On the other hand, if the amount thereof exceeds 25 at%, the FCC phase is stabilized. Hence, the amount of Ni falls in the range of 13 to 25 at%.

[0024] Outside of the scope of the invention, if the amount of at least one selected from among molybdenum (Mo) and aluminum (Al), which is a component that may substitute for cobalt (Co), is less than 13 at%, the FCC phase is stabilized. On the other hand, if the amount thereof exceeds 25 at%, the BCC phase is stabilized. Hence, the amount thereof preferably falls in the range of 13 to 25 at%.

[0025] Outside of the scope of the invention, if the amount of manganese (Mn), which is a component that may substitute for nickel (Ni), is less than 13 at%, the BCC phase is stabilized. On the other hand, if the amount thereof exceeds 25 at%, the FCC phase is stabilized. Hence, the amount thereof preferably falls in the range of 13 to 25 at%.

30 [0026] Outside of the scope of the invention, an interstitial element such as C or N in a metal alloy is subjected to interstitial solid solution in the metal matrix to thus enhance the strength of the alloy due to solid-solution strengthening effects during metal deformation. When at least one element of C and N is added in an amount of 1 at% or more based on the total at% of the alloy, the FCC phase is stabilized. In order to utilize the effect of deformation-induced phase transformation by inducing the metastable FCC phase, it is preferred that the above element be added in an amount of less than 1 at%.

[0027] The inevitable impurities are components other than the above alloying elements and are unavoidable components that are inevitably incorporated into the alloying elements or during the production process.

[0028] The medium-entropy alloy may be composed of a metastable FCC phase or a combination of a metastable FCC phase and a BCC phase at room temperature. Here, it is preferred that the fraction of the metastable FCC phase be high in the interests of improvements in tensile strength and elongation. The fraction of the metastable FCC phase is preferably 50% or more. However, the fraction of the metastable FCC phase is not necessarily 50% or more.

[0029] Also, the medium-entropy alloy may have tensile strength of 500 MPa or more and elongation of 50% or more at room temperature (298 K).

45 [0030] Also, the medium-entropy alloy may have tensile strength of 1000 MPa or more and elongation of 40% or more at a cryogenic temperature (77 K).

[Examples 1 to 4]

#### Production of medium-entropy alloy

50 [0031] First, Co, Cr, Fe, and Ni metals having purity of 99.9% or more were prepared.

[0032] The metals thus prepared were weighed in the mixing fractions shown in Table 1 below.

## EP 3 660 178 B1

[Table 1]

	Metal mixing fraction (at%)			
	Co	Cr	Fe	Ni
Example 1	17.50	10.00	55.00	17.50
Example 2	16.25	10.00	57.50	16.25
Example 3	15.00	10.00	60.00	15.00
Example 4	13.75	10.00	62.50	13.75

**[0033]** The metals prepared in the above fractions were placed in a crucible, heated to 1550°C and thus melted, and then cast into 150 g of an alloy ingot having a cuboid shape having a width of 33 mm, a length of 80 mm, and a thickness of 7.8 mm, using a mold.

**[0034]** In order to remove the oxide formed on the surface of the cast alloy, surface grinding was performed. The thickness of the ground ingot was 7 mm.

**[0035]** The surface-ground ingot having a thickness of 7 mm was subjected to homogenization heat treatment at 1100°C for 6 hr and then cold rolling to a thickness from 7 mm to 1.5 mm. Furthermore, the cold-rolled alloy plate was annealed at 800°C for 10 min.

[Comparative Examples 1 and 2]

### Production of alloy for Comparative Examples

**[0036]** The alloys of Comparative Examples were manufactured in the same manner as in Examples using the components in the amounts shown in Table 2 below.

[Table 2]

	Metal mixing fraction (at%)			
	Co	Cr	Fe	Ni
Comparative Example 1	14.50	5.00	66.00	14.50
Comparative Example 2	12.50	10.00	65.00	12.50

**[0037]** The alloy ingot was cast in the same manner as in Examples, followed by homogenization heat treatment at 1100°C for 6 hr and then cold rolling to a thickness from 7 mm to 1.5 mm in the same manner as in Examples.

**[0038]** Furthermore, the cold-rolled alloy plate was annealed at 800°C for 10 min in the same manner as in Examples.

### Component analysis results

**[0039]** The actual components of the alloys manufactured in Comparative Examples 1 and 2 and Examples 1 to 4 after annealing treatment were analyzed using EDS. The results are shown in Table 3 below.

[Table 3]

	EDS analysis composition (at%)			
	Co	Cr	Fe	Ni
Comparative Example 1	14.34	5.10	66.29	14.27
Example 1	17.37	10.52	55.58	16.53
Example 2	16.16	10.21	57.41	16.22
Example 3	14.54	10.68	60.89	13.89
Example 4	13.55	10.27	62.55	13.63
Comparative Example 2	12.23	10.81	65.31	11.65

[0040] As is apparent from Table 3, the actual composition falls slightly out of the range of the initial metal mixing fractions, but may be regarded as almost the same level considering the purity of metals and impurities which may be incorporated during the production process. All of Examples fell in the composition range of the medium-entropy alloy according to the present invention, comprising 6 to 12.5 at% of Cr, 55 to 62.5 at% of Fe, 13 to 25 at% of Co, and 13 to 25 at% of Ni.

XRD analysis results

[0041] FIG. 1 shows the results of XRD measurement at room temperature of the annealed alloys of Comparative Examples 1 and 2 and Examples 1 to 4.

[0042] XRD measurement was performed after grinding in the order of sandpaper Nos. 600, 800 and 1200 and then electrolytic etching in 8% perchloric acid in order to minimize phase transformation due to deformation during grinding of a test specimen.

[0043] As shown in FIG. 1, Comparative Example 1 was composed of the BCC phase, Examples 1 to 4 were composed mainly of the metastable FCC phase, and Comparative Example 2 was composed mainly of the BCC phase and included a small amount of the FCC phase.

[0044] Specifically, when the amount of Fe was increased and the amounts of Co and Ni were decreased, the stability of the FCC phase was deteriorated, and consequently the metastable FCC phase was formed in Examples 1 to 4. In Comparative Examples 1 and 2, in which Fe was added in an amount of 65 at% or more, the FCC phase was no longer in the metastable state but became unstable, and the BCC phase was relatively stabilized.

Tensile test results

[0045] The results of tensile testing at room temperature (298 K) and at a cryogenic temperature (77 K) of the annealed alloys of Comparative Examples 1 and 2 and Examples 1 to 4 according to the present invention are shown in FIGS. 2 and 3 and Table 4 below.

[0046] FIGS. 2 and 3 are graphs showing the results of tensile testing at room temperature and at a cryogenic temperature, respectively, in which the horizontal axis designates the engineering strain and the vertical axis designates the engineering stress. Based on the graphs of the test results, the results of analysis of physical properties such as yield strength, tensile strength and elongation of Comparative Examples and Examples 1 to 4 are given in Table 4 below.

[Table 4]

Test specimen	Room temperature			Cryogenic temperature (77 K)		
	Yieldstrength (MPa)	Tensilestrength (MPa)	Elongation (%)	Yieldstrength (MPa)	Tensilestrength (MPa)	Elongation (%)
Comparative Example 1	850	975	24	1336	1455	33
Example 1	280	550	68	615	1024	126
Example 2	274	568	86	543	1164	118
Example 3	226	534	98	526	1508	82
Example 4	228	787	67	620	1649	47
Comparative Example 2	579	996	26	1110	1516	30

[0047] As is apparent from FIGS. 2 and 3 and Table 3, the tensile properties at room temperature of the medium-entropy alloys of Examples 1 to 4 according to the present invention exhibited yield strength of 226 to 280 MPa, tensile strength of 534 to 787 MPa, and elongation of 67 to 98%.

[0048] Furthermore, excellent tensile properties at a cryogenic temperature, such as yield strength of 526 to 620 MPa, tensile strength of 1024 to 1649 MPa, and elongation of 47 to 126%, were manifested. In contrast, the tensile properties at room temperature of the medium-entropy alloys of Comparative Examples 1 and 2 were as follows: since the initial crystal structure was mostly composed of a BCC structure, there were neither strengthening effects nor elongation enhancement effects due to deformation-induced phase transformation between tensile deformation at room temperature and tensile deformation at a cryogenic temperature, and tensile yield strength and tensile strength were high at room temperature and at a cryogenic temperature due to the BCC structure, but elongation was low, resulting in brittleness.

**[0049]** In particular, the alloy of Example 3, including a large amount of the FCC phase in the metastable state, manifested excellent tensile properties at a cryogenic temperature, such as yield strength of 526 MPa, tensile strength of 1508 MPa, and elongation of 82%, which were not previously reported.

#### 5 Deformation-induced phase transformation

**[0050]** FIG. 4 shows the analytical results of EBSD for phase transformation of the medium-entropy alloy of Example 3 during deformation at room temperature and at a cryogenic temperature according to the present invention.

10 **[0051]** As shown in FIG. 4, the alloy of Example 3 included a very small amount of the BCC phase and was composed mainly of the metastable FCC phase before deformation, and the fraction of the BCC phase was remarkably increased after deformation at room temperature (298 K) and at a cryogenic temperature (77 K). In particular, phase transformation from the FCC phase into the BCC phase occurs over the entire region after deformation at a cryogenic temperature, and this phase transformation contributes greatly to the improvement of cryogenic mechanical properties, as shown in FIG. 3.

15 **[0052]** Therefore, with regard to the cryogenic mechanical properties, the fraction of the FCC phase before deformation is preferably set to 50% or more.

[Table 5]

	Before deformation (vol%)	After deformation (298 K) (vol%)	After deformation (77 K) (vol%)
20 Comparative Example 1	91.26	93.96	98.99
25 Example 1	0.34	15.07	28.46
Example 2	0.38	20.26	36.12
Example 3	0.41	27.68	56.71
Example 4	25.68	62.23	85.08
30 Comparative Example 2	87.81	89.20	94.87

35 **[0053]** Table 5 shows the results of ferritescope measurement of the BCC phase fraction (vol%) of the alloys of Comparative Examples 1 and 2 and Examples 1 to 4 according to the present invention, before deformation and after deformation at room temperature and at a cryogenic temperature.

40 **[0054]** As is apparent from Table 5, the alloys of Examples 1 to 3 included a small amount of the BCC phase before deformation and were increased in the fraction of the BCC phase due to phase transformation between deformation at room temperature and deformation at a cryogenic temperature. Also, the alloy of Example 4 was relatively increased in BCC phase stability compared to the alloys of Examples 1 to 3, and thus it was confirmed that 25.68 vol% of the BCC phase was included before deformation and that the fraction of the BCC phase was increased due to phase transformation between deformation at room temperature and deformation at a cryogenic temperature. The alloys of Comparative Examples 1 and 2 were very high in BCC phase stability compared to the alloys of Examples 1 to 4, and thus it was confirmed that 91.26 at% and 87.81 vol% of the BCC phases, respectively, were included before deformation and that the fraction of the BCC phase was increased due to phase transformation between deformation at room temperature and deformation at a cryogenic temperature.

#### Claims

50 1. A medium-entropy alloy, consisting of:

6 to 12.5 at% of Cr,

55 55 to 62.5 at% of Fe,

13 to 25 at% of Co,

13 to 25 at% of Ni, and

a remainder of inevitable impurities, wherein deformation-induced phase transformation from a face-centered cubic (FCC) phase into a body-centered cubic (BCC) phase occurs upon plastic deformation, wherein the

## EP 3 660 178 B1

medium-entropy alloy is composed of a combination of a body-centered cubic BCC phase and a metastable face-centered cubic FCC phase, or is composed of a metastable face-centered cubic FCC phase alone.

2. The medium-entropy alloy of claim 1, wherein a fraction of the metastable FCC phase is 50% or more.

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### Patentansprüche

1. Eine mittel-entropische Legierung, bestehend aus:

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6 bis 12,5 at% Cr,  
55 bis 62,5 at% des Fe,  
13 bis 25 at% von Co,  
13 bis 25 at% Ni, und

15

einen Rest unvermeidlicher Verunreinigungen, wobei eine verformungsinduzierte Phasenumwandlung von einer kubisch-flächenzentrierten (FCC) Phase in eine kubisch-körperzentrierte (BCC) Phase bei plastischer Verformung auftritt, wobei die mittelentropie Legierung aus einer Kombination einer kubisch-körperzentrierten BCC-Phase und einer metastabilen kubisch-flächenzentrierten FCC-Phase besteht oder nur aus einer metastabilen kubisch-flächenzentrierten FCC-Phase besteht.

20

2. Mittelentropie Legierung nach Anspruch 1, wobei der Anteil der metastabilen FCC-Phase 50 % oder mehr beträgt.

### Revendications

25

1. Un alliage à entropie moyenne, constitué de:

6 à 12,5 at% de Cr,  
55 à 62,5 at% de Fe,  
13 à 25 at% de Co,  
13 à 25 % atomique de Ni, et

30

un reste d'impuretés inévitables, dans lequel une transformation de phase induite par déformation d'une phase cubique à faces centrées (FCC) en une phase cubique à corps centré (BCC) se produit lors d'une déformation plastique, dans lequel l'alliage d'entropie moyenne est composé d'une combinaison d'une phase BCC cubique à corps centré et d'une phase FCC cubique à faces centrées métastable, ou est composé d'une phase FCC cubique à faces centrées métastable seule.

35

2. Alliage d'entropie moyenne selon la revendication 1, dans lequel une fraction de la phase FCC métastable est de 50% ou plus.

40

45

50

55

FIG. 1

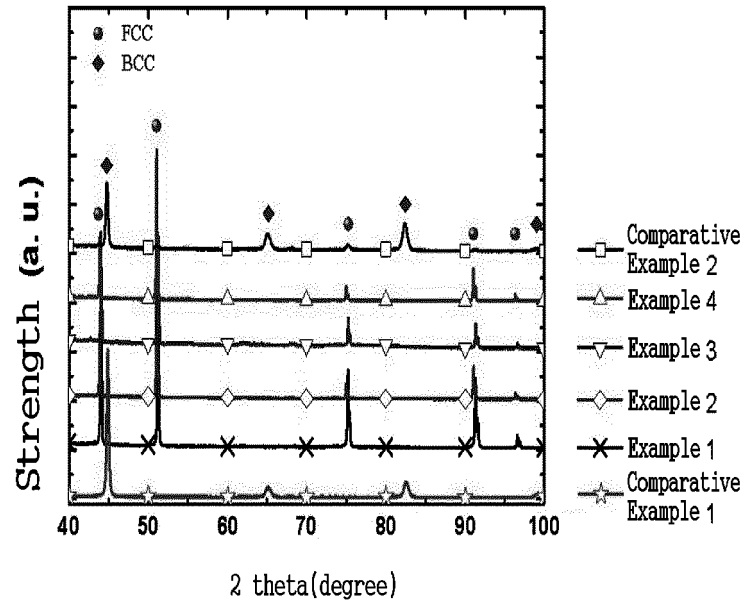


FIG. 2

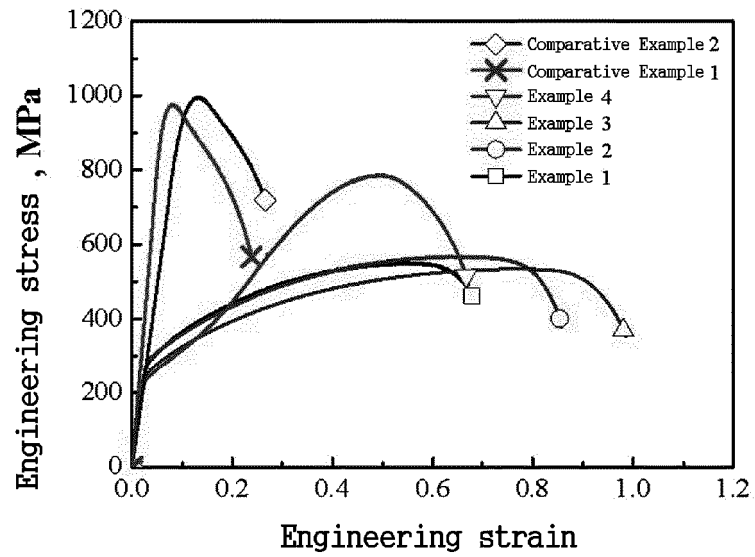


FIG. 3

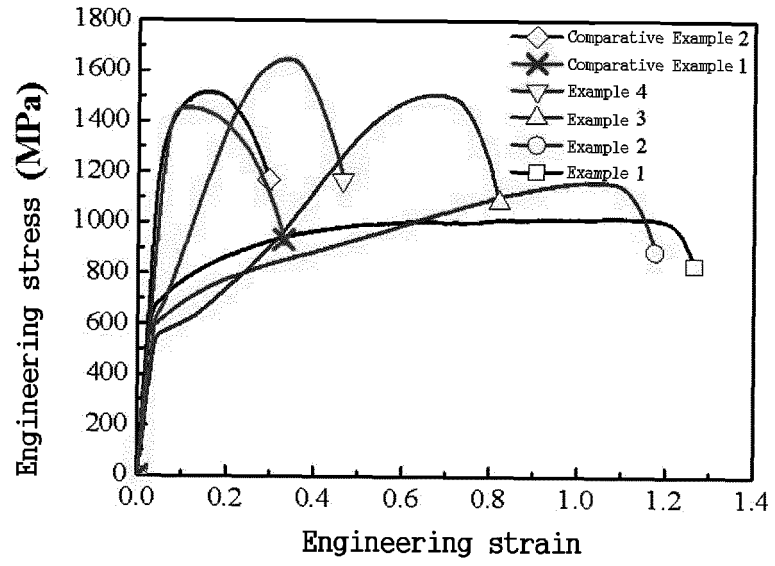
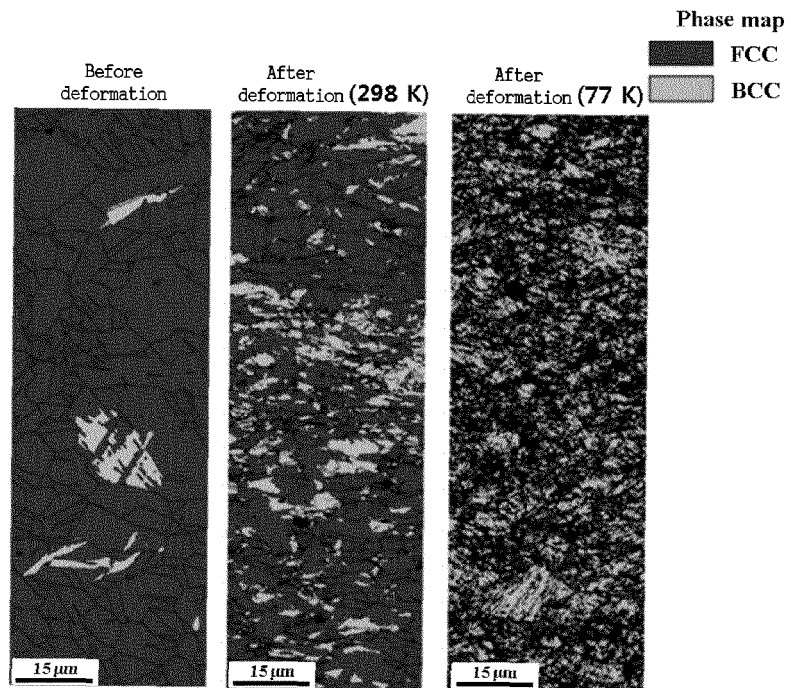


FIG. 4



**REFERENCES CITED IN THE DESCRIPTION**

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