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CRYSTALLOGRAPHIC FEATURES OF THE VARIOUS PHASES OF TI 6WT%A1 2WT%NB-1WT%MO (T1 6211) AS A FUNCTION OF THERMAL HISTORY

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ABSTRACT

An investigative study to establish the crystallographic features of various phases in Ti 6w%A1-2wt%Nb-1wt%Ta-1wt%Mo Ti 6211 as a function of thermal history was carried out.
As received Ti6211 alloy samples were equilibrated at 1025 degrees centigrade in argon atmosphere and down-quenched to 900, 700, 600 and 500 degrees centigrade and then isothermally annealed for 48 hours and quenched to room temperature. X-ray analysis and X-ray diffraction studies were conducted to identify the various phases and their stability at various times and temperatures.
Based on these studies it was found that quenching in 900, 700 and 800 degrees centigrade, isothermal annealing and finally quenching to room temperature produced $\alpha + \beta$ structure. Isothermal annealing at both 600 and 500 degrees centigrade produced ($\alpha + \alpha''$) and ($\alpha + \alpha''$) structures respectively. While quenching directly to room temperature from 1025 degrees centigrade after isothermal annealing produced martensitic $\alpha' + \alpha''$.
The β-phase in all these cases was of course retained β and the α-phase could be equilibrium α or martensitic α'. Based on the X-ray diffractin of the as-quenched alloy samples a tentative partial phase diagram for Ti 6211 was suggested. Crystallographic parameters of the various phases were also calculated.

KEYWORDS: Crystallography, x-ray diffraction, titanium alloy, martensites, heat treatment

1.0 INTRODUCTION

Ti 6211 has been developed for possible marine application requiring thick sections. The microstructure of a mear aspha or $\alpha + \beta$ alloys such as Ti 6211 has a grat influence on various mechanical properties such as strength, ductility and fracture toughness. $\alpha + \beta$ alloys generally (Boradaile, and Jeal, 1981) have low cycle fatigue properties because of fine-grained mixture of $\alpha + \beta$ structure with relatively high proportion of primary alpha. Depending on the heat treatment in the $\alpha + \beta$ field metastable beta-phase can be retained (Williams, 1980) in Ti 6211 alloy. A possibility exists that such metastable beta phase will transform martensitically under service load. James and moon (1970) have discussed the effects of stress-induced martensite on the mechanical on the mechanical properties of three Titanium alloys.

Two martensites detected by (Williams, et. al., 1981), alpha (hexagonal) and alpha double prime (orthorhombic) were reported for Titanium alloys, with similar chemistry to Ti6211. However no detailed study of the extent, formation and stability of the phases at various times and temperatures has been done and their crystallographic parameters were speculative. An investigation of these aforementioned features would be accomplished in the present study.

Pure Titanium element, commonly called electrolytic titanium is one of the most important non-ferrous metals because of the wide application of its alloys in hostile environments, military, aerospace and marine industries.

Pure titanium has a low temperature α-phase of hexagonal close-packed crystal structure, with lattice parameters (a = 0.295nm), c = 0.468nm and c/a = 1.587) and high temperature β-phase of body-centered cubic crystal structure with lattice parameter, a = 0.332 at 900 degree centigrade (Donald, Hultgren 1973).

The $\alpha \longrightarrow \beta$ transformation temperature is 882 degree centigrade. Its melting point is 1668 ± 10 degree centigrade and its boiling point is estimated to be 3260 degree centigrade.

Commercial applications of titanium are limited by the ingress of interstitial alloying elements into the titanium matrix like oxygen, nitrogen, carbon and hydrogen. In fact at high temperatures titanium is like a porous sponge into which the above alloying elements readily permeate.

1.1 Titanium alloys:

Titanium alloys properly neat-treated have unique applications is very severe conditions. They exhibit high creep, fatigue and corrosion resistance in very hostile environments both at high and low temperatures, good high strength/high toughness potential endearing the alloys to special military applications (Borradaile, and Jeal, 1980).

Titanium alloys are classified into the following structural types, depending on the alloying elements, namely; α or near α, α - β and β or near β titanium alloys. α-Titanium alloys result from α-stabilizing alloying elements, namely; (Al, Ge, Ga, C, O and N) and β-Titanium alloys result from β-stabilizing elements, namely; (V, Mo, Ta, Nb). The proportionate addition of both α-stabilizers and β-stabilizers results in the production of α - β titanium alloys (Donald and Hultgren, 1973).

2.0 Objective of the Experimental Investigation

The objective of this investigation was to establish the crystallographic features of the various phases in Ti 6wt% Al-2wt.% Nb – 1wt.% - Ta – 1wt.% Mo, henceforth called Ti6211) as a function of thermal history. Ti 6211 was newly developed to optimize the properties of titanium alloys for possible applications in marine structures, steam turbines, aerospace industries, anodes for copper recycling, pollution control systems, medical prosthesis, deep diving submersibles, desalination, nuclear power plants, corrosion resistant chemical process equipment, conveyor belts etc. (Obikwelu, 2003).

Ti 6211 was composed of both α- and β-stabilizers and the stability of the various phases at various temperatures are not known. (Borradaile and Jeal, 1980) have noted that mechanical properties were impaired by certain phases in the Titanium alloys.

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Hence the present study would establish the crystallographic features of the various equilibrium and metastable phases that existed in Ti 6211 after isothermal heat treatments at various temperatures.

3.0 Materials and Methods

3.1 Materials and Equipment

(5 x 2 x2) cm^3 rectangular blocks of Ti-6wt.% Al-2wt.%Nb - 1wt.%Ta - 1wt.%Mo(Ti 6211), coaxial furnace system, X-ray diffractometer, metallographic units, argon cylinder, temperature potentiometer, digital millivoltmeter.

Sample A (samples heat-treated at 500 degrees centigrade)
Sample B (samples heat-treated at 600 degrees centigrade)
Sample C (samples heat-treated at 700 degrees centigrade)
Sample D (samples heat-treated at 800 degrees centigrade)
Sample E (samples heat-treated at 900 degrees centigrade)

3.2 Methods

3.2.1 The as-received sample plate was cut into small rectangular blocks (5 x 2 x 2) cm^3 and polished in 120-grade abrasive to remove blade marks and flatten the surface. More polishing was done through 600 grit before slightly hot-working the sample at about 1090°C. The samples were air-cooled and profusely polished to remove any alpha case and oxidized layer. The clean samples of final dimensions (3 x 1 x1)cm^3 were subsequently polished to 600 grit and then heat-treated in the specially designed furnace.

The samples were solution-treated in the upper furnace stage for about 35 minutes at 1030°C and step-quenched to the lower furnace stage and annealed at various temperatures for 48 hours. These annealing temperatures at this lower furnace stage were measured by means of a potentiometer and a digital millivoltmeter so that the circuit was completed by remotely marching a leg switch. Pure titanium getters were used inside the system to further minimize the absorption of interstitial elements like hydrogen and oxygen into the alloy.

3.2.2 Heat Treatment

Titanium alloys at high temperatures absorb especially oxygen at an alarming rate, therefore a conventional furnace was inadequate for heat treating Ti alloys.

A special coaxial two-stage furnace system bathed with argon was designed. In addition, quenching within seconds and within the furnace system under the inert argon atmosphere was accomplished by looping an electrical resistor with the sample so that the circuit was completed by remotely marching a leg switch. Pure titanium getters were used inside the system to further minimize the absorption of interstitial elements like hydrogen and oxygen into the alloy.

3.2.3 X-ray Diffraction

X-ray diffraction was done on the heat-treated samples to identify the phases and index the planes at the various annealing temperatures. Each sample was re-polished on a rotating wheel with cloth using 0.05micron alumina and then etched slightly for 15 seconds in Kroll's etchant (95cc H_20, 3.5cc HNO_3 and 1.5cc HF).

4.0 Result and Discussion

4.1 After the solution treatment and annealing of all the samples of Ti6211 at various temperatures, X-ray diffraction techniques was used to identify the stable phases at each temperature after 48 hours annealing time.

Detailed analysis of the x-ray results was conducted and the summaries for 600°C, 800°C and 900°C are presented on Tables 1 -3.

Table 1: Experimental 2θ angles for Sample E annealed at 900°C for 48 hours in argon atmosphere and quenched in water. Data taken at 22°C with Cu Kα radiation.

Peak #	2θ (deg)	Hk1) or (hki1)	Phase identif
1	35.38	$(10\bar{1}0)$	α
2	38.24	(0002)	α
3	39.51	$(\bar{1}10)$	β
4	40.14	$(10\bar{1}1)$	α
5	52.89	$(10\bar{1}2)$	α
6	62.96	$(11\bar{2}0)$	α
7	71.60	(211)	β
8	74.64	$(20\bar{2}0)$	α
9	76.46	$(11\bar{2}2)$	α
10	81.41	(0004)	α
11	85.00	(220)	β
12	106.25	$(21\bar{3}0)$	β
13	111.00	$(21\bar{3}1)$ α, (222) β	α, β

Table 2: Experimental 2θ angles for Sample D annealed at 800°C for 48 hours in argon atmosphere and quenched in water. Data taken at 22°C with Cu Kα radiation.

Peak#	2θ (deg)	(hkl) or (hkil)	Phase Identification
1	35.38	$(10\bar{1}0)$	α
2	38.58	(0002)	α
3	39.52	(110)	β
4	40.48	$(10\bar{1}1)$	α
5	53.20	$(10\bar{1}2)$	α
6	57.12	(200)	β
7	63.50	$(11\bar{2}0)$	α
8	71.0	$(10\bar{1}3)$.	α
9	71.64	(211)	β
10	74.64	$(20\bar{2}0)$	α
11	76.45	$(11\bar{2}2)$	α
12	77.77	$(20\bar{2}1)$	α
13	82.21	(0004)	α
14	84.53	(220)	β
15	86.85	$(20\bar{2}2)$	α
16	102.78	$(20\bar{2}3)$	α
17	106.19	$(21\bar{3}0)$	α
18	111.60	$(21\bar{3}1)_\alpha$, $(222)_\beta$	α, β

Table 3: Experimental 2θ angles for Samples B annealed at 600°C for 48 hours in argon atmosphere and quenched in water. Data taken at 22°C with Cu Kα radiation.

Peak#	2θ (deg)	(hkl) or (hkil)	Phase Identificatio
1	35.31	$(10\bar{1}0)$	α
2	38.60	$(0002)_\alpha$	α
3	39.73	$(111)_{\alpha''}$,	α''
4	40.28	$(10\bar{1}1)$	α
5	53.11	$(112)_{\alpha''}$, $(10\bar{1}2)_\alpha$	α'', α
6	63.23	$(11\bar{2}0)$	α
7	71.90	$(10\bar{1}3)_\alpha$, $(113)_{\alpha''}$	α, α'',
8	76.53	$(11\bar{2}2)$, (221)	α, α''
9	77.73	$(20\bar{2}1)$	α
10	82.21	(0004)	α
11	85.50	$(222)_{\alpha''}$	α'',
12	106.71	$(21\bar{3}0)$	α
13	111.90	$(21\bar{3}1)$	α
14	115.06	$(11\bar{2}4)$	α

Based on the sample annealed at 700°C and quenched in water, average lattice parameters for the HCP phase were calculated to be

$a_\alpha = 2.938A^\circ$
$c_\alpha = 4.670\ A^\circ$

These values agreed with literature on lattice parameters for titanium alloy systems (Boarradiale and Jeal, 1980)
Similarly the lattice parameters for the β -phase (BCC crystal structure) and those of the martensitic orthorhombic phase calculated from samples annealed respectively at 800°C and 500°C and quenched in water, thus:
Lattice parameters for β-phase (BCC)

$a_\beta = 3.225A^\circ$, were consistent with literature values.

The orthorhombic phase parameters, calculated thus:
$a_\alpha = 3.040A^\circ$
$b_\alpha = 4.944\ A^\circ$
$c_\alpha = 4.637\ A^\circ$
were consistent with literature (Murakami, 1980).

(note that $1A^\circ = 10^{-10}m$).

Based on these lattice parameters, a complete set of allowed reflections for the hcp and BCC phases were calculated using CuK_α radiation. The typical results of these calculations were summarized in Table 4 and 5 with increasing 2θ angles.

Table 4: Possible reflections of the BCC (β-phase) in Ti6211 Data taken from Sample D annealed 48 hours in argon atmosphere at 800°C and quenched in water at room temperature. Cu K_α radiation was used.

$a_o = 3.225$Å			2θ (in deg.) (Cu K_α)		
	hk1	**dhk1**	λ	λ_1	λ_2
	110	2.280	39.525	39.490	39.593
	200	1.612	57.140	57.089	57.244
	211	1.316	71.719	71.651	71.856
	220	1.140	85.101	85.014	85.270
	310	1.019	98.321	98.211	98.540
	222	0.930	111.981	111.840	112.26
	321	0.861	127.113	126.923	127.49

Table 5: Possible reflections of the HCP (α,α') in Ti6211 Data taken from Sample D annealed 48 hours in argon atmosphere at 700°C and quenched in water at room temperature. Cu K_α radiation was used.

a = 2.9378A
c = 4.6697A

HCP		2θ (in deg.) (CuK_α)*		
hkil	dhkl	λ	λ_1	λ_2
$10\bar{1}0$	2.544	35.279	35.249	35.340
0002	2.334	38.573	38.540	38.640
$10\bar{1}1$	2.234	40.373	40.339	40.443
$10\bar{1}2$	1.720	53.257	53.210	53.352
$11\bar{2}0$	1.468	63.356	63.298	63.473
$10\bar{1}3$	1.327	71.966	70.966	71.170
$20\bar{2}0$	1.272	74.611	74.539	74.756
$11\bar{2}2$	1.243	76.662	76.587	76.812
$20\bar{2}1$	1.227	77.849	77.772	78.002
0004	1.167	82.691	82.607	82.858
$20\bar{2}2$	1.117	87.287	87.196	87.468
$10\bar{1}4$	1.061	93.203	93.103	93.404
$20\bar{2}3$	0.985	103.009	102.890	102.248
$21\bar{3}0$	0.961	106.682	106.555	106.937
$21\bar{3}1$	0.941	110.020	109.889	110.292
$11\bar{2}4$	0.913	115.211	115.061	115.510

* λ = Weighted average of the wavelengths of an unresolved K doublet of Cu radiation.
λ_1 = Wavelength of CuK_{α_1} radiation

A typical x-ray diffraction pattern for Ti6211, solution-treated and annealed respectively at 500°C, 600°C, 700°C, 800°C and 900°C for 48 hours in argon atmosphere is shown in Fig. 1. it was not possible to distinguish between α_o and α' in the x-ray diffraction work alone since they have similar crystal structures and very nearly the same lattice parameters.
Thus, all the hcp reflections in this study were designated alpha, α

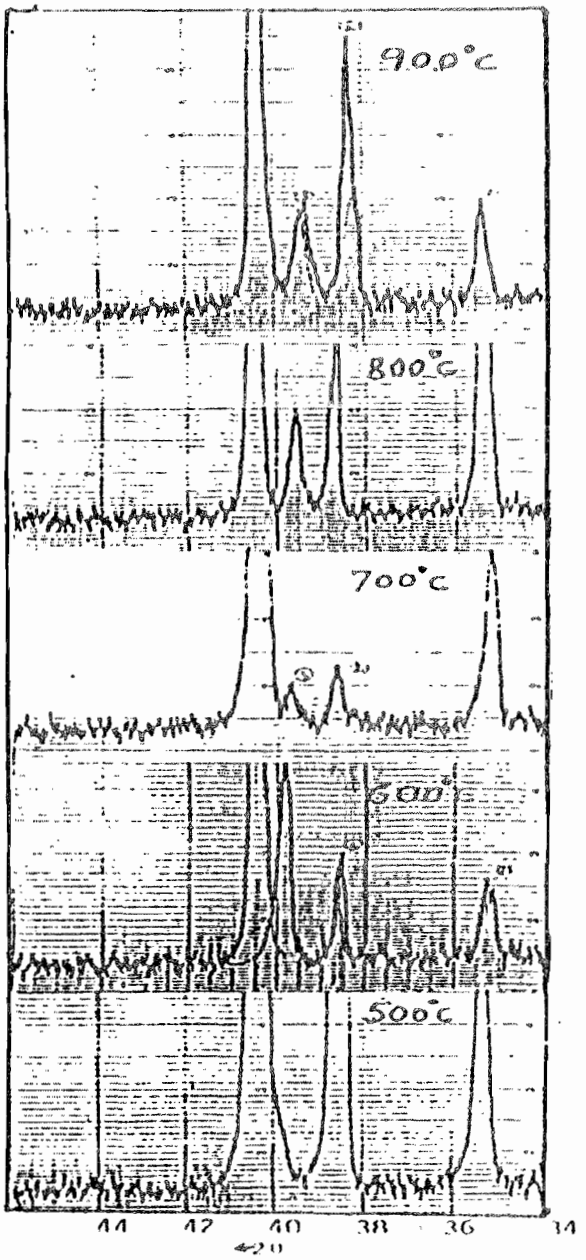

Fig. 1: X-ray Diffraction Pattern of Samples A, B, C, D and E under investigation

4.2 **Stable Phases in Ti6211**

The high temperature β-phase and the low temperature α-phase were known to occur in pure Titanium (Seagle, 1968) This was confirmed in the present study (at 500 and 600 degree centigrade, equilibrium α_o and α" were detected

The α-stabilizing alloying elements retain α -phase at low temperatures while the β-stabilizing alloying elements retain the β-phase at low temperature (Murkerjee, et. al., 1984) and because of this, some traces of β-phase were retained from high temperature quenching in the present study.

Quenching the high temperature β-phase to below M_s temperature generated the following phase transformations (Murakami, 1980):

i. $\longrightarrow \beta\ \alpha' + \alpha''$

ii. $\longrightarrow \omega_{ath} + \beta_r$

where α' = hexagonal Ti martensite
α" = orthorhombic Ti martensite
ω_{ath} = arthermal omega phase
β_r = beta phase retained.

Retained β -phase might decompose thus (Murakami, 1980):

$\beta_r \longrightarrow \alpha + \beta$

Or through compound precipitation thus:

$\beta_r \longrightarrow$ ordered $\alpha_2 + \beta$

$\beta_r \longrightarrow$ ordered $\beta_2 + \beta$

These transformations were confirmed in the present study except that α_2 β_2 and ω_{ath} were not detected in the present study, probably because of the sensitivity of the X-ray unit (the interference of the background radiation).

4.3 **Crystallography of α' and α" Martensites:**

The Burger's mechanism for β(BCC) $\longrightarrow$ α'(hcp) transformation in Titanium alloys (Burger, W. G., 1931) was also confirmed in the present study.

The formation of the second martensite (orthorhombic phase α") and the α'(hcp) in Titanium alloys observed in the present study can be explained in terms of beta-stabilizing element composition and simple atomic shuffling postulated by (Murkerjee 1984).

4.4 **Splitting of α peaks in X-ray pattern**

The splitting of the α peaks on the X-ray diffraction pattern observed by (Sasano, et al, 1980) with increasing beta-stablizing molybdenum content in TiAl was confirmed at 500° and 600° where α" was detected

5.0 **Conclusion**

From the X-ray analysis, it was found that the phases formed in Ti6211 alloy studied were α_o, α', α" and β

α_o, α' and β retained were identified in the samples annealed at 900°C, 800°C and 700°C, and quenched in water at room temperature, with the β-phase decreasing in volume fraction as the annealing temperatures were lowered. For samples annealed at 600°C and 500°C and quenched in water at room temperature, the phases identified were α_o and α"

From the study, the crystal lattice parameters of the prevalent phases in Ti6211 were found to be as follows

β-phase (BCC):
$a_\beta = 3.225A^\circ$ (3.225×10^{-10}m)
α-phase (HCP):
$a_\alpha = 2.938A^\circ$ (2.938×10^{-10}m)
$c_\alpha = 4.670A^\circ$ (4.670×10^{-10}m)
Orthorhombic phase
$a_\alpha = 3.040A^\circ$ (3.040×10^{-10}m)
$b_\alpha = 4.944A^\circ$ ($4\ 944 \times 10^{-10}$m)
$c_\alpha = 4.637A^\circ$ (4.637×10^{-10}m)
($1A^\circ = 10^{-10}$m)

These values compared favorably with lattice parameters of titanium alloy systems in the literature (Murakami, 1980)

Based on this study, a schematic α and β phase boundaries in the Ti6211 alloy system was developed (Fig. 2) and the problem of devising a partial phase diagram for Ti6211 was solved.

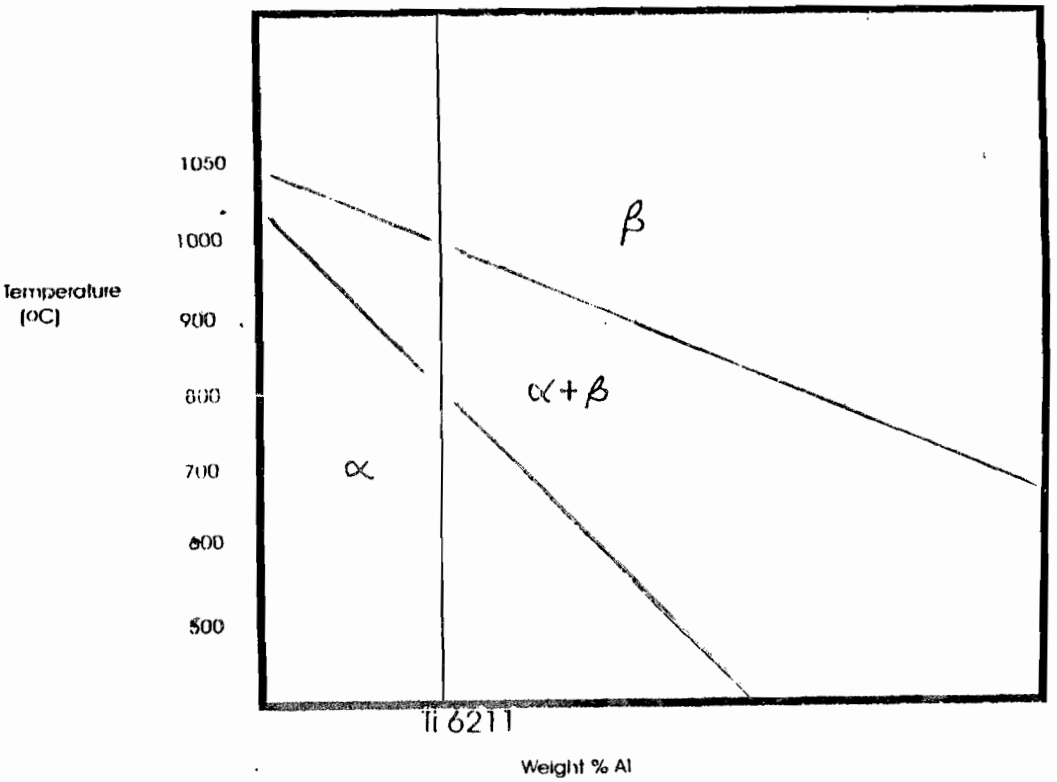

Fig. 2: Schematic α and α + β boundaries resulting from Samples A, B, C, D and E (under investigation)

The detection of retained β- phase in this alloy system would be useful in material alloy design involving titanium alloys because there is the possibility of stress-induced transformation of this phase in service and the consequent effects on the mechanical properties as found by (James and Moon, 1970).

6.0 REFERENCES

Buckman, R. W. Jnr., 2000 New Applications for Tantalum and Tantalum alooys. Journal of Metals, Vol. 52. (3), 32.

Burgers, W. G., 1931. Physica. Gravi. 1. 561.

Borradaile, J. T and Jeal, R. H., 1980. Titanium'80 Science and Technology. Proceedings of the international conference on Titanuim, Kyoto, Japan 1980 ed. H. Kimura and O. Izumi, AIME Publication, 1, p 141

Murakami, Y, James, D. W and Moon, D. W., 1980. Titanium '80 Science and Technology, proceedings of the international conference on Titanium Kyoto, Japan 1980 ed. Kimura. H. and Izumi. O. AIME Publication, 1, p 141.

Murkerjee, K. Kato, M. and Obikwelu, D O N., 1984 Martensitic Transformation in Titanium Alloys: A Generalized Approach, 5th International Conference on Titanium, 10 Sept 1984 Germany

Obikwelu. D. O N., 2003 Materials in the service of man Paper presented at the Technical Meeting of the Nigerian society of Engineers, Warri. Nigeria.

Sasano, H., Williams, J C., Donald, T and Hultgren. R Titanium '80 science and Technology, proceedings of the International conference on Titanium, Kyoto, Japan 1980 ed. Kimura H. and Izumi, O AIME Publication 1 p 717

Seagle, S. R., 1968. Metals Engineering Institute The American society of Metals, U. S A