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# Development of mechanical properties of structural high-carbon low-alloy steels through modified heat treatment

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The modified heat treatment, which produces a mixed structure of martensite and lower bainite through short-term isothermal transformation at just above the martensitic transformation temperature, $M_s$ temperature, followed by oil quenching (after conventional austenitization), has been applied to three high-carbon low-alloy steels with different levels of nickel and chromium contents at similar molybdenum levels, in which carbon was allowed to replace relatively expensive additions of nickel and chromium, for their ultra-high strength application. The significant conclusions are as follows: an ultra-high strength steel of 1900 MPa yield-stress grade with a high toughness level can be obtained when about 60 vol% lower bainite is associated with 473 K tempered martensite of 0.60% C-1.80% Ni-0.80% Cr-0.25% Mo steel. If approximately 25 vol% lower bainite appears in 673 K tempered martensite of the steel, a 1700 MPa yield-stress grade steel with high toughness and moderate ductility levels can be attained. However, alloying nickel is essential to some extent for development of the mechanical properties with the modified heat treatment suggested in the present work.

## 1. Introduction

Commercial, ultra-high strength, low-alloy steels, i.e. AISI 4340 and BS En 24 with 0.2% yield stress which is in excess of 1400 MPa, has currently been used in aircraft and high-performance aerospace systems. So far, considerable effort has been devoted to improve the mechanical properties by metallurgical techniques such as heat treatments or compositional modifications. For example, improvement in the mechanical properties through the high austenitizing treatment, and the importance of mechanical stability of retained austenite with compositional modifications, have been reported [1–7].

Recently there has been increased demand for ultra-high strength applications of low-alloy steels with high carbon contents in which the required mechanical properties and hardenability may be achieved by allowing carbon to replace the relatively expensive additions of nickel and chromium. This aims to economize a cost of materials. It is well known that strength values of martensitic steels are significantly enhanced by an increase of carbon content. However, increases in strength are usually associated with corresponding decreases in the toughness values which then limit the extent of structural applications of such steels. A better approach to overcome this problem is to develop the mechanical properties of the steels by microstructural control through modified heat-treatment techniques. We have provided one potential solution to this problem by developing 0.40% C–Ni–Cr–Mo ultra-high strength steel (AISI 4340 or BS En 24 grade) having a mixed structure of martensite and lower bainite, which is produced by short-term isothermal transformation just above its martensitic transformation temperature, $M_s$, followed by water or oil quenching (after conventional austenitization) [8]. The effectiveness of the mixed structure in improving the mechanical properties can be attributed to the fact that lower bainite, which appears in acicular form and partitions prior austenite grains, effectively causes a refinement of the martensitic substructure (i.e. lath width and packet diameter) which leads to increased strength and that the lower bainite provides a significant increased resistance to the steel which would normally cause brittle failure. Based on these experiments, it is expected that such a microstructure would permit improvement in the mechanical properties of high-carbon low-alloy steels where brittleness has been an obstacle to widespread use.

In the present work, three high-carbon low-alloy steels with different levels of nickel and chromium have been studied to determine the effect of the modified heat treatment, which produces a mixed structure of martensite and lower bainite through short-term isothermal transformation at just above the $M_s$ temperature followed by oil quenching (after conventional austenization), on the mechanical properties of high-carbon structural low-alloy steels for ultra-high strength application.

## 2. Experimental procedure

The chemical composition and $M_s$ temperatures of the steels used in this investigation are shown in Table I. The steels were received as 130 mm diameter hot-rolled

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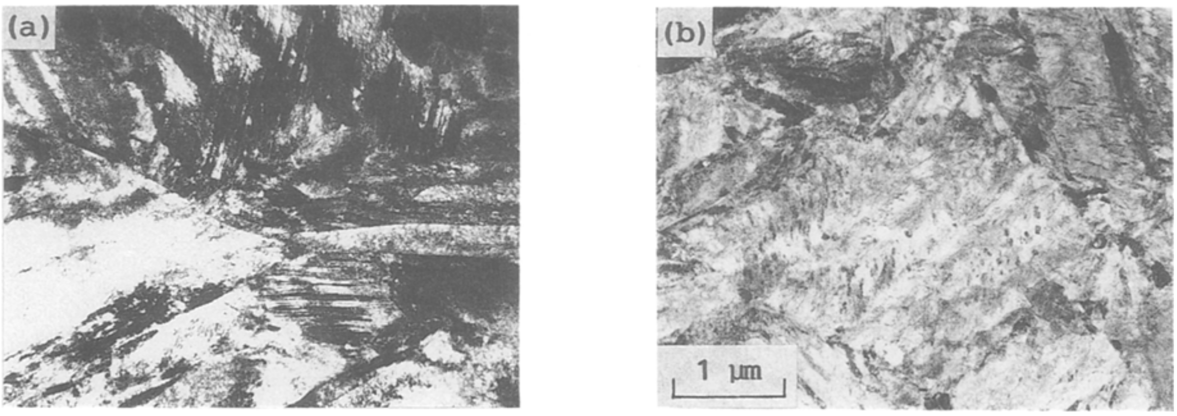

*Figure 1* Representative transmission electron micrographs of steel A having (a) fully martensitic and (b) bainitic structures.

bar stock. Test steels were cut from the bars with their longitudinal axes parallel to the rolling direction and machined to the required dimensions. Each was fully annealed. In order to obtain a mixed structure of martensite and lower bainite, and a fully lower bainitic structure, the test steels were quenched directly into a lead–tin bath followed by isothermal transformation at a temperature just above their $M_s$ temperatures ($M_s$ temperature plus 10 K) from 280 sec to 72 ksec after austenitization at 1133 K for 7.2 ksec in an argon atmosphere tube furnace with a flat zone temperature accuracy of $\pm 0.5$ K. The steels were subsequently oil quenched. The lead–tin bath used in this investigation had a sufficiently large thermal capacity. The fully martensitic steels were produced by heat treatment followed by direct oil quenching after austenitization at 1133 K for 7.2 ksec. Tempering was done in an oil bath held at 473 K or a lead bath kept at 673 K for 7.2 ksec. Mechanical properties were determined through smooth and notch tensile tests. Smooth tensile specimens with a gauge length of 25 mm and a cross-section 1.5 mm × 4.0 mm and notch tensile test-pieces with a 2 mm V-notch in both sides and an effective cross-section of 1.5 mm × 4.0 mm were used. The tensile tests were performed with an Instron machine at ambient temperature (293 K) and at a constant strain rate of $3.35 \times 10^{-4}\,\text{sec}^{-1}$. The microstructure was categorized by optical metallography and thin-foil transmission electron microscopy (TEM). Specimens for optical metallography were first etched with a supersaturated aqueous solution of picric acid containing 0.2 wt % of a surface agent, which is known to reveal the prior austenite grain size [8, 9]. Then, the specimens were etched in 2% nitral solution to delineate the bainitic structure. Quantitative metallographic determination of the volume fraction of the second-phase lower bainite was performed using the standard point counting [10]. Thin foils were prepared from 1.5 mm thick heat-treated specimens, first by mechanically grinding to a 0.1 mm thickness, then chemically thinning in a mixed solution of hydrofluoric acid and hydrogen peroxide, and finally, electropolishing in a mixed solution of phosphoric and chromic acids.

## 3. Results and discussion

### 3.1. Microstructural identification

The substructure identification of martensite and bainite attained using a short-term isothermal transformation at temperature just above the $M_s$ temperature (after austenitization at 1133 K) has been made using thin-foil TEM. Fig. 1 shows the typical transmission electron micrographs of fully martensitic and bainitic structure for steel A. The TEM revealed that lath martensite, involving relatively large amounts of twinned plates, occurred by direct oil quenching after austenitization at 1133 K (Fig. 1a). Isothermal transformation at 523 K led to typically lower bainite which consisted of ferrite plates in which large amounts of internal fine carbide precipitates were orientated in one direction across the plates (Fig. 1b). Optical metallography revealed that when the lower bainite was associated with the martensite, it appeared in acicular form and partitioned the prior austenite grains of the parent martensite (Fig. 2a). So, in order to estimate quantitatively the above microstructural features, the relationship between the volume fraction of lower bainite ($V_B$) or martensitic size partitioned by the lower bainite was established, the results are given in Table II. Between 200 and 300 lower bainites were taken at random to measure the average lower bainitic width ($B_W$) and length ($B_L$). The average martensitic size ($M_S$) was determined by linear intercept measurements on optical micrographs.

The results obtained are summarized as follows: (1) $B_W$ has a value (1.0 to 1.3 $\mu$m) nearly independent of $V_B$; (2) $B_L$ shows the amount of order of prior austenite grain size (9.5 to 9.9 $\mu$m) irrespective of $V_B$; (3) $M_S$ decreases from 6.7 to 3.6 $\mu$m as $V_B$ increases from 10

TABLE I Chemical composition (wt %) and $M_s$ temperatures of steels

| Designation of steel | C | Si | Mn | P | S | Ni | Cr | Mo | $M_s$(K)* |
|---|---|---|---|---|---|---|---|---|---|
| A | 0.60 | 0.27 | 0.80 | 0.012 | 0.019 | 1.80 | 0.80 | 0.25 | 513 |
| B | 0.60 | 0.28 | 0.78 | 0.015 | 0.020 | 1.08 | 0.83 | 0.24 | 518 |
| C | 0.60 | 0.23 | 0.82 | 0.013 | 0.021 | – | 1.41 | 0.24 | 523 |

* $M_s$ temperature was determined by standard dilatometry.

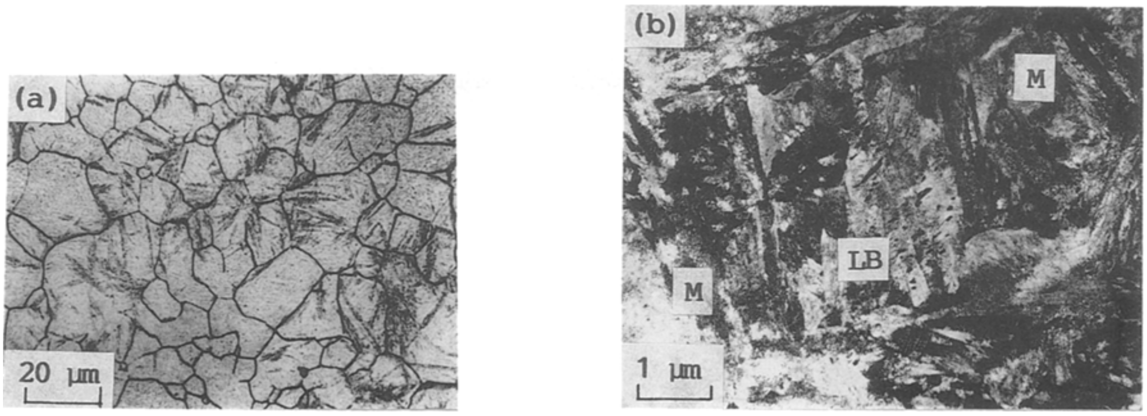

*Figure 2* Representative optical and thin-foil transmission electron micrographs of steel A having a mixed structure of martensite and 25 vol % lower bainite. M and LB indicate martensite and lower bainite, respectively.

to 75 vol %. Furthermore, thin-foil TEM revealed that lower bainite associated with martensite causes a refinement of lath width as well as packet size, as shown in Fig. 2b. However, bainitic substructure in the mixed structure is somewhat similar to that of a single-phase bainite, as can be seen from a comparison of Figs 1b and 2b.

## 3.2. Mechanical properties

Mechanical properties of steels A having a mixed structure of martensite and lower bainite are shown in Tables III and IV. Noteworthy is the fact that isothermal transformation of lower bainite provided a better combination of strength and notch toughness when lower bainite at 50 vol % and above was associated with tempered martensite at 473 K, while unstable brittle fracture occurred if lower bainite below 50 vol % appeared in martensite tempered under the same conditions (Table III). In particular, with 60 vol % lower bainite, 0.2% yield stress ($\sigma_y$), ultimate tensile strength ($\sigma_u$), total elongation and true notch tensile strength (TNTS), in which the fracture load in the notch tensile test was divided by the cross-sectional area at fracture, of the steels, achieved 1910.6 to 1920.5 MPa, 2140.4 to 2180.3 MPa, 2.6 to 2.8% and 2280.5 to 2310.9 MPa, respectively.

Another important fact is that the desirable effect on the mechanical properties became evident at 25 vol % lower bainite if the steels were tempered at 673 K. With the lower bainite, $\sigma_y$, $\sigma_u$, total elongation and TNTS of the steels attained values of 1742.3 to 1750.2 MPa and 1820.3 to 1825.9 MPa, 4.0 to 4.2% and 2250.5 to 2310.3 MPa, respectively (Table IV).

These improvements in the mechanical properties, as discussed in detail in terms of metallographical observations and the law of mixture from previous papers [8], could be explained by the fact that the second-phase lower bainite causes a refinement of the martensitic substructure, i.e. lath width and packet diameter, and that, in addition, the lower bainite provided increased resistance to brittle fracture. However, with below 50 vol % lower bainite, unstable fracture occurred and consequently decreased its strength and toughness. In order to elucidate this reason, metallographic observations of initiating microcracks were made through the sections adjacent to the fracture surface of the notched specimen having a lower bainite content below 50 vol %, by scanning electron microscopy (SEM). SEM revealed that the secondary cracks were found with martensite area or in the vicinity of the two-phase interfaces, as can be seen in Fig. 3. This fracture profile contrasts with observations in a previous paper [8], that when lower bainite is associated with 473 K tempered martensite, microcracks being initiated at one lower bainite are stopped at another lower bainite, resulting in improved toughness. This difference could be caused by the distinction between contents of the twinned martensite in two steels, i.e. the deterioration of the mechanical properties can be attributed to the fact that the lower bainite cannot fully relieve stress concentration on brittle martensite which contains more twinned martensite when the steels have a lower bainite content below 50 vol % [11, 12].

## 3.3. Effect of nickel content on mechanical properties

For the purpose of saving expensive nickel alloying, the effect of nickel content on the mechanical properties has been investigated using steels B and C, which contain combinations of the elements found in the more economical alloys. The nickel content of steel B is about 0.90 wt % less than that for steel A at similar chromium level. For steel C, the required hardening

TABLE II Microstructural parameters of mixed structure of martensite and lower bainite

| Volume fraction of lower bainite, $V_B$(%) | Lower bainitic size | | Martensitic size, partitioned, $M_S$($\mu$m) |
|---|---|---|---|
| | Width, $B_W$($\mu$m) | Length, $B_L$($\mu$m) | |
| 10 | 1.0 | 9.5 | 6.7 |
| 25 | 1.1 | 9.7 | 5.2 |
| 50 | 1.2 | 9.7 | 4.5 |
| 75 | 1.3 | 9.9 | 3.6 |

TABLE III Room-temperature mechanical properties in 473 K tempered condition of steels A having mixed structure of martensite and lower bainite

| Volume fraction of lower bainite (vol %) | 0.2% yield stress, $\sigma_y$ (MPa) | Ultimate tensile strength, $\sigma_u$ (MPa) | Total elongation (%) | True notch tensile strength, TNTS (MPa) |
|---|---|---|---|---|
| 0 | 1830.2 | 1880.6 | not evaluated | 1380.6 |
| | 1845.2 | 1870.5 | not evaluated | 1350.5 |
| 10 | 1890.5 | 1910.6 | not evaluated | 1460.3 |
| | 1910.3 | 1930.3 | not evaluated | 1400.2 |
| 25 | 1901.7 | 1940.2 | not evaluated | 1410.2 |
| | 1912.3 | 1930.5 | not evaluated | 1480.5 |
| 50 | 1900.3 | 2040.5 | 1.8 | 2040.3 |
| | 1910.3 | 2020.4 | 1.9 | 2004.8 |
| 60 | 1910.6 | 2140.4 | 2.8 | 2280.5 |
| | 1920.5 | 2180.3 | 2.6 | 2310.9 |
| 75 | 1860.5 | 2000.6 | 3.2 | 2250.6 |
| | 1852.3 | 1996.3 | 3.1 | 2268.0 |
| 90 | 1742.6 | 1948.2 | 3.1 | 2220.7 |
| | 1760.2 | 1960.1 | 3.3 | 2280.6 |
| | 1660.0 | 1880.5 | 4.0 | 2250.6 |
| 100 | 1648.2 | 1850.3 | 4.2 | 2263.5 |

has been attained by using chromium and molybdenum as the main alloying elements without adding expensive nickel. The results are shown together with those of steel A in Tables V and VI. It can be seen from these tables that there is little difference between the mechanical properties of steels A and B, independent of tempering conditions. However, the mechanical properties in the 473 K tempered condition of steel C having a mixed structure of martensite and 60 vol % lower bainite, were significantly inferior to those for steels A and B (Table V), while there was little difference between the mechanical properties in the 673 K temperature condition (Table VI). SEM observations through the section adjacent to fracture surface revealed that when lower bainite was associated with 473 K tempered martensite of steel C, the secondary cracks were found within the martensite area or in the vicinity of the two-phase interfaces even for specimens having lower bainite content above 50 vol % (Fig. 4).

In this regard, Jolley has systematically studied the toughening effect of nickel at lower temperature on Fe and Fe–C alloys [13, 14]. He has demonstrated that addition of nickel to the alloys significantly improves the lower temperature toughness. This occurs because nickel directly increases the lower temperature intrinsic toughness of the ferrite matrix. This has been proved by the fact that nickel alloyed with iron exhibits wavy slip when strained at temperatures down to at least ~ 50 K; whereas, unalloyed iron changes from wavy to planar slip when the test temperature is lowered to ~ 120 K.

On the basis of the above results and literature, the beneficial effect of nickel addition on the mechanical properties is thought to be due to the fact that alloying nickel exerts an effect on the toughness of the mixed structure of martensite and lower bainite, by changing the intrinsic toughness of the parent martensite. That is, this could be due to the fact that alloying nickel increases the intrinsic toughness of the parent martensite through facilitating the cross-slip during deformation. These results also suggest that alloying nickel is essential to some extent for development in the mechanical properties of low alloy steels with high carbon contents through the modified heat treatment suggested in the present work.

## 4. Conclusions

The modified heat treatment, which produces a mixed structure of martensite and lower bainite through short-term isothermal transformation just above the $M_s$ temperature followed by oil quenching (after

TABLE IV Room-temperature mechanical properties in 673 K tempered condition for steels A having mixed structure of martensite and lower bainite

| Volume fraction of lower bainite (vol %) | 0.2% yield stress, $\sigma_y$ (MPa) | Ultimate tensile strength, $\sigma_u$ (MPa) | Total elongation (%) | True notch tensile strength, TNTS (MPa) |
|---|---|---|---|---|
| 0 | 1630.2 | 1723.0 | 2.3 | 1750.6 |
| | 1620.5 | 1728.2 | 2.5 | 1790.3 |
| 10 | 1700.6 | 1785.3 | 3.8 | 2150.3 |
| | 1720.3 | 1800.0 | 3.7 | 2010.6 |
| 25 | 1750.2 | 1820.3 | 4.2 | 2250.5 |
| | 1742.3 | 1825.9 | 4.0 | 2310.3 |
| 50 | 1640.3 | 1740.3 | 4.3 | 2300.6 |
| | 1630.2 | 1740.2 | 4.5 | 2286.9 |
| 60 | 1630.2 | 1728.3 | 4.0 | 2210.6 |
| | 1610.3 | 1720.2 | 4.3 | 2259.3 |
| 75 | 1600.3 | 1680.6 | 4.0 | 2200.5 |
| | 1590.6 | 1650.6 | 4.4 | 2230.2 |
| 100 | 1550.3 | 1630.2 | 4.2 | 2240.3 |
| | 1530.6 | 1621.6 | 4.4 | 2250.6 |

*Figure 3* Scanning electron micrograph of secondary microcracks (indicated by arrows) observed in a section adjacent to the fracture surface of 2 mm V-notched steel A having 25 vol % lower bainite (tempered at 473 K).

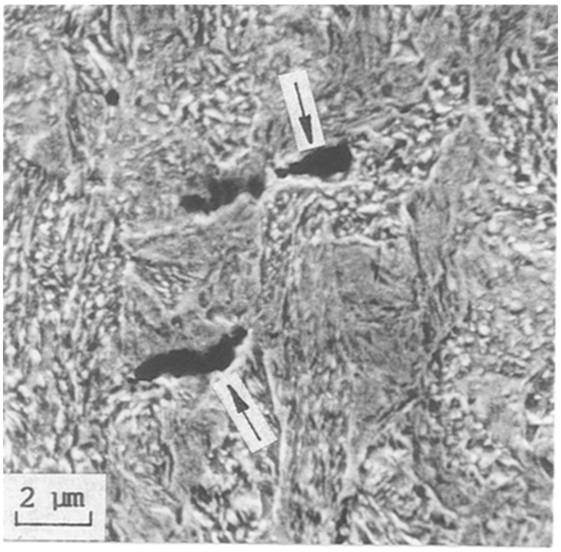

*Figure 4* Scanning electron micrograph of secondary microcracks (indicated by arrows) observed in a section adjacent to the fracture surface of 2 mm V-notched steel C having 60 vol % lower bainite.

conventional austenitization), has been applied to develop the mechanical properties of three high-carbon, low-alloy steels with different levels of nickel or chromium contents at similar molybdenum level, and for their ultra-high strength applications. It has been shown that the ultra-high strength steel of 1900 MPa yield-stress grade with a high toughness level can be obtained when about 60 vol % lower bainite is associated with 473 K tempered martensite of 0.60% C–1.80% Ni–0.80% Cr–0.25% Mo steel. If approximately 25 vol % lower bainite appears in 673 K tempered martensite of the steel, 1700 MPa yield-stress grade steel with high toughness and moderate ductility levels can be attained. It is also shown that alloying nickel is essential to some extent for development of the mechanical properties with the modified heat-treatment suggested in the present work.

## References

1. G. Y. LAI, W. E. WOOD, R. A. CLARK, V. F. ZACKAY and E. R. PARKER, *Met. Trans.* **5** (1974) 1663.
2. W. E. WOOD, *Engng Fract. Mech.* **7** (1975) 219.
3. R. O. RITCHIE, B. FRANCIS and W. L. SERVER, *Met. Trans. A* **7A** (1976) 831.
4. R. O. RITCHIE and R. M. HORN, *ibid.* **9A** (1978) 331.
5. K. H. KHAN and W. E. WOOD, *ibid.* **9A** (1978) 899.
6. B. V. N. RAO and G. THOMAS, *ibid.* **11A** (1980) 441.
7. W. SARIKAYA, B. G. STEINBERG and G. THOMAS, *ibid.* **13A** (1982) 2227.
8. Y. TOMITA and K. OKABAYASHI, *ibid.* **14A** (1983) 485.
9. *Idem, ibid.* **17A** (1986) 1203.

TABLE V Comparison between room-temperature mechanical properties in 473 K tempered condition of steels A, B and C having mixed structure of martensite and 60 vol % lower bainite

| Designation of Steel | 0.2% yield stress, $\sigma_y$ (MPa) | Ultimate tensile strength, $\sigma_u$ (MPa) | Total elongation (%) | True notch tensile strength, TNTS (MPa) |
|---|---|---|---|---|
| A-1 | 1910.6 | 2140.4 | 2.8 | 2280.5 |
| A-2 | 1920.5 | 2180.3 | 2.6 | 2310.9 |
| B-1 | 1900.8 | 2040.5 | 1.9 | 2140.0 |
| B-2 | 1920.6 | 2020.4 | 2.2 | 2180.9 |
| C-1 | 1810.5 | 1810.5 | not evaluated | 1380.5 |
| C-2 | 1826.4 | 1830.1 | not evaluated | 1390.2 |

TABLE VI Comparison of room-temperature mechanical properties in 673 K tempered condition of steels A, B and C having mixed structure of martensite and 25 vol % lower bainite

| Designation of Steel | 0.2% yield stress, $\sigma_y$ (MPa) | Ultimate tensile strength, $\sigma_u$ (MPa) | Total elongation (%) | True notch tensile strength, TNTS (MPa) |
|---|---|---|---|---|
| A-1 | 1750.2 | 1820.3 | 4.2 | 2250.5 |
| A-2 | 1742.3 | 1825.9 | 4.0 | 2310.3 |
| B-1 | 1760.5 | 1825.9 | 3.8 | 2280.5 |
| B-2 | 1730.8 | 1823.8 | 4.1 | 2210.6 |
| C-1 | 1730.2 | 1825.3 | 3.5 | 2190.6 |
| C-2 | 1750.6 | 1840.2 | 4.0 | 2160.2 |

10. J. E. HILLIARD and J. W. CAHN, *Trans. TMS AIME* **221** (1961) 344.
11. K. OKABAYASHI, Y. TOMITA and I. KUROKI, *Tetsu-to-Hagane* **62** (1976) 661.
12. *Idem*, *ibid*. **62** (1976) 991.
13. W. JOLLEY, *Trans. TMS AIME* **242** (1968) 306.
14. *Idem*, *J. Iron Steel Inst.* **206** (1968) 170.

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