

EVALUATION OF THE INFLUENCE OF INTERCRITICAL TREATMENT VARIABLES ON THE TRANSFORMATION BEHAVIOUR OF A CARBON STEEL

BY

*** K. K. ALANEME and **C. M. KAMMA**

* Department of Metallurgical and Materials Engineering
Federal University of Technology, Akure Nigeria
Late Professor of Metallurgical and Materials Engineering
University of Lagos Akoka, Nigeria

ABSTRACT

This research study was focused on determining the intercritical treatment conditions for optimized martensite and ferrite formation for enhanced strength and plasticity in plain carbon steels. As-received plain carbon steel of predetermined composition was used as test material. Ferrite/pearlite structure was produced for use as initial microstructure before cold rolling to achieve varied degrees of deformations. Intercritical anneals at 740^o, 760^o, and 780^oC for various holding times before quenching in water was subsequently performed on the samples. The transformation behaviour was studied with the aid of optical microscopy and hardness curves obtained by plotting hardness values against log of soaking time. From the results it is observed that the structural evolution when intercritical annealing is carried out at 740^oC and 760^oC, is characterized by one

mode and four transformation stages; while two transformation modes consisting of five and three transformation stages, were observed when treatment is carried out at 780^oC. Schematic models were used to explain the mechanism of each stage of transformation observed.

Keywords: *intercritical annealing; transformation behaviour; optical microscopy; models; Strength.*

1.0 INTRODUCTION

In the utilization of steel based materials for various applications, Materials and Metallurgical Engineers are always conscious of recommending the steel grade that will perform efficiently in service. The prospect of obtaining such highly efficient steel grade is realized through adoption of aspects of alloying and phase transformations, which help in the development and control of microstructures

(Martin et al, 1997; Rios et al, 2005). This is made possible because the different mechanisms of phase transformations control the formation of structures, especially structure – sensitive properties of materials (Gorelik, 1981; Doherty et al., 1997).

In steel, microstructure development involving the exploitation of phase transformation has been observed to be problematic due to competing phase reactions like precipitation, recrystallization and crystallographic phase changes; especially when treatment is carried out within the ($\alpha + \gamma$) phase region (Alaneme, 2007; Stuwe et al., (2002); Kamma and Hornbogen, 1984). This has made it impossible to maximise the advantages of treatment in the ($\alpha + \gamma$) phase region, which potentially can help induce high strength and plasticity in carbon steel: a property combination which is of great consideration in the development of automobile and truck body parts; and sheet metals with high formability at high strength levels for fabrication purposes.

In this present work an attempt is made to study the phase transformation behaviour in carbon steel when treatment is carried out in the ($\alpha + \gamma$) phase region: in order to establish

optimum thermo mechanical conditions for optimised mechanical properties.

2.0 MATERIALS AND METHODS

The material for the investigation is a plain carbon steel as-supplied in rectangular size of 20cm x 14cm x 6 cm. Chemical analyses to determine the composition of the steel was performed spectrometrically. The chemical composition (in Wt %) is as follows: C (0.32); Si (0.26); Mn (0.89); P (0.0401); S (0.0021); Cr (0.109); Ni (0.035); Mo (0.0042); V (0.0012).

The as- supplied steel material was subjected preliminarily to machining to produce samples of strip configurations having thickness ranging from 2.5mm to 10mm. Thereafter normalizing treatment to annul the thermal and mechanical history of the steel; and also to induce a starting point (initial) microstructure in the steel was carried out. The normalizing treatment was carried out by holding the steel material at 870⁰C for one hour in a muffle furnace and then cooling in air. Thereafter cold rolling operation was adopted to plastically deform all the samples to a uniform thickness of 2mm (80% deformation). The deformed samples were later cut into specimen sizes of 10mm x 20mm x 2mm.

After the cold rolling operation, the cut test pieces were set aside for the intercritical thermal treatment. The test pieces were treated at intercritical temperatures of 740°C , 760°C , and 780°C ; and were held for 0, 1, 5, 15, 30 and 60 minutes; followed by quenching rapidly in water.

The hardness tests were performed using a Rockwell Hardness Tester. The hardness measurements were used to establish the transformation behaviour by studying graphical plots of the evolution of hardness with time of intercritical treatment. The specimens for the hardness tests were ground and polished to obtain a smooth surface. To increase the accuracy of readings, measurements were taken at three points on each specimen and the average of these determined. The results of the hardness – intercritical treatment time plots are shown in figures 1, 3 – 4.

Microstructural examination was utilised to follow the transformation process. The test specimens were prepared for the microscopic analysis using standard methods for grinding and polishing. Etching of the samples was carried out with the use of 2% Nital solution, which was applied on the surface of each test samples for 10 seconds, and then rinsed quickly in water.

Photomicrographs of the examined structures were taken thereafter.

3.0 Results and Discussion

3.1 Intercritical Annealing of the Normalised Samples Treated at 740°C

Figure 1 shows the plot of hardness against annealing time for the normalised samples treated at 740°C . Examination of the plot shows a pattern (trend) which suggests that during annealing of these samples; the overall transformation progressed in four stages within the time duration of 60 minutes. These stages are shown in Figure 2. The four stages have been indicated as follows:

Stage I – characterized by a slight drop in hardness, indicative of recovery processes without any structural change; Stage II – characterized by a sharp drop in hardness, indicative of intense spheroidization of cementite followed by recrystallization of ferrite;

Stage III – characterized by a sharp rise in hardness, indicative of the formation of prior austenite which forms martensite on quenching in water; and Stage IV – characterized by another drop in hardness, indicative of grain growth. However, the

drop occurs at a much higher hardness value.

From Figure 1, it is observed that stage I transformation ended after 1 minute of annealing for all samples irrespective of percent deformation. The drop in hardness experienced at this stage is due to recovery brought about by stress relieving of the as-deformed structure (comparing Plate 1 and Plate 2). Stage II transformation occurred between 1-5 minutes of annealing for the specimens subjected to 20% and 50% deformation; while for the heavily deformed specimens (70% and 80% deformed specimens), stage II was extended to about 15 minutes of annealing. The significant drop in hardness experienced in stage II is attributed to decomposition of pearlite and primary recrystallization of the ferrite phase (Plate 3); emanating from the intense annihilation of the high dislocation density induced in the material by cold rolling – leading to significant reduction of the stored energy in the material (Rios et al., 2005). Stage III transformation which has caused an increase in hardness, is observed to end after 30 minutes annealing for specimens subjected to high levels of prior deformation (50%, 70% and 80% deformation). Thus peak hardness values appeared after 30

minutes annealing – marking the end of stage III. However stage III transformation is observed to be uncompleted even after 60 minutes annealing for the 20% deformed sample. This implies that longer annealing time will be needed to attain peak hardness for this low deformation level. The increase in hardness observed in stage III is as a result of martensite formation resulting from the quenching in water of the prior austenite nucleated during the intercritical treatment (Plate 4). The quantity of prior austenite nucleated during annealing, and consequently the fraction of martensite increased with holding time (Cota et al., 2003). Further holding into stage IV caused a reduction in hardness from the peak value.

The decrease in hardness at this stage is attributed to the growth of recrystallized α / γ grains before quenching (Plate 5). This increase in grain size is due to the drive to reduce the overall strain energy of the structure by reduction of the total phase boundaries (Torres et al., 2002). Thus it can be drawn that optimum martensite and ferrite formation for enhanced hardness properties (strength) can be achieved when treatment is carried out within stage III.

3.2 Effects of Intercritical Annealing at 760°C on Transformation Behaviour

Figure 3 shows the hardness - time profile of all experimental test samples treated at 760°C. Examination of these plots show the same general transformation trend as observed during treatment at 740°C: this is with respect to the 4 – stage transformation model developed.

From the plot it is seen that stage I transformation ended after 1 minute of treatment for all deformed samples as in the case of treatment at 740°C (Figure 2).

Stage II transformation is observed to be completed after 5 minutes annealing for all deformed samples. However, it is observed that the decrease in hardness – characterizing stage II transformation – was smaller than that obtained in the case of treating at 740°C. This is due to the increased decomposition and diffusion rate which facilitates γ nucleation when the treatment is performed at relatively higher intercritical temperatures (Cota et al., 2003). Stage III transformation is observed to commence after 5 minutes of annealing for all deformed samples and ends after 15 minutes of annealing for the heavily deformed samples (70% and 80% deformed samples). Thus annealing for greater than 15

minutes does not bring about much remarkable increase in hardness unlike the case of the 20% and 50% deformed samples in which it is observed that stage III transformation is uncompleted even after 60 minutes of annealing: indicating that peak hardness will only be attained by annealing for longer periods in excess of 60 minutes. Thus selection of annealing periods greater than 15 minutes for the heavily deformed samples (70% and 80% deformation) will be influenced by other desired properties like ductility and low yield ratio. It is noteworthy to appreciate that the general increase in hardness is due to the increased volume fraction of Martensite (formed from quenching the prior austenite in water) which forms at 760°C in comparison to 740°C (Mazinani and Poole, 2007).

3.3 Effects of Intercritical Annealing at 780°C on Transformation Behaviour

Figure 4 shows the hardness – time profiles for all normalised samples treated at 780°C. From the plot it is observed that the profiles are different from those covered for annealing at 740°C and 760°C (Figures 1 and 3). In this intercritical annealing temperature of 780°C, the transformation process seemed to be done in five stages for

the normalised specimens that underwent 20% and 50% prior deformation while those that were subjected to 70% and 80% deformation showed a 3 – stage transformation mode. Both of these transformation modes are shown schematically in Figures 5 and 6 respectively.

At low deformations (20 and 50%) the curves suggest that the overall transformation progresses in five stages (Figure 5). The five stages are:

Stage I – stress relief (recovery), characterised by a slight drop in hardness; Stage II – recrystallization of both α and γ particularly from the pearlite, characterised by a sharp rise in hardness; Stage III – grain growth of recrystallized α grains which is competing with further γ nucleation; causing arrest in increase in hardness; Stage IV – further γ nucleation and homogenization causing further increase in hardness due to more martensite formation; and Stage V – formation of larger α and γ grains the balance of which has resulted in loss of hardness.

At higher deformations (70 and 80%) the curves show that the overall transformation progresses in three stages (Figure 6). The three stages are:

Stage I – stress relief, characterised by a slight drop in hardness; Stage II – recrystallization of both α and γ accompanied with homogenization of γ , causing increased hardenability and more martensite formation, and characterised by increase in hardness; and Stage III – grain growth of α and γ characterised by drop in hardness.

Thus at low prior deformations, peak hardness is obtained when treatment is carried out at stage IV, while for high prior deformations, peak hardness is obtained when treatment is carried out at stage II of the overall transformation process.

CONCLUSIONS

This research study was focused on determining the intercritical treatment conditions for optimized martensite and ferrite formation for enhanced strength and plasticity in plain carbon steels. From the results obtained, the following conclusions are drawn:

For all cold deformed initial microstructures, the process of producing duplex structures of ferrite and martensite when intercritical annealing is carried out at 740°C and 760°C, is characterised by four transformation stages. However when intercritical

annealing is carried out at 780°C , the transformation stages are for low percent deformations modified to five; while for heavier deformations prior to during intercritical annealing at 780°C , the transformation leading to the production of duplex structures was characterized by three stages. The three transformation modes observed: one (four stage transformation) for treatment at 740°C and 760°C , and two

(five stage and three stage transformation) for annealing at 780°C ; were developed to transformation models which helped in explaining the transformation behaviour for each treatment condition and determination of process conditions required for the production of duplex microstructures (consisting of ferrite and martensite) with optimized mechanical properties.

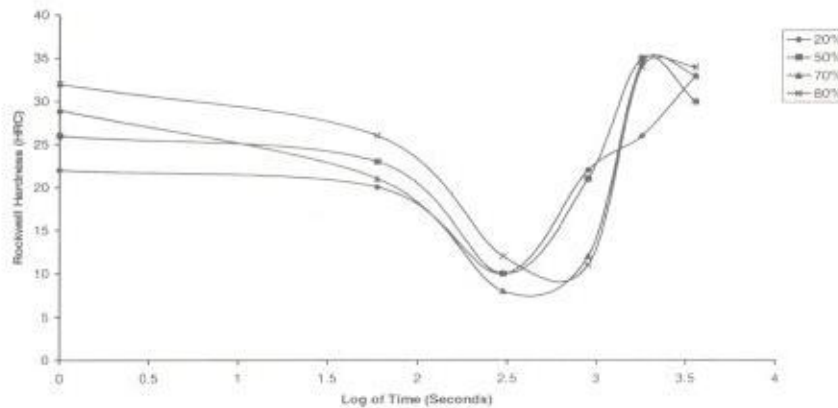


Figure 1: Rockwell Hardness Vs Annealing Time Plot For Normalized Specimens Treated at 740°C

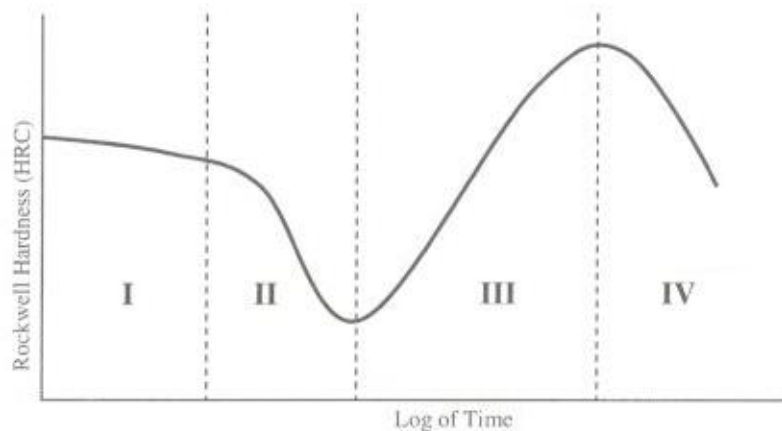


Figure 2: Schematic representation of the 4-Stage transformation model.

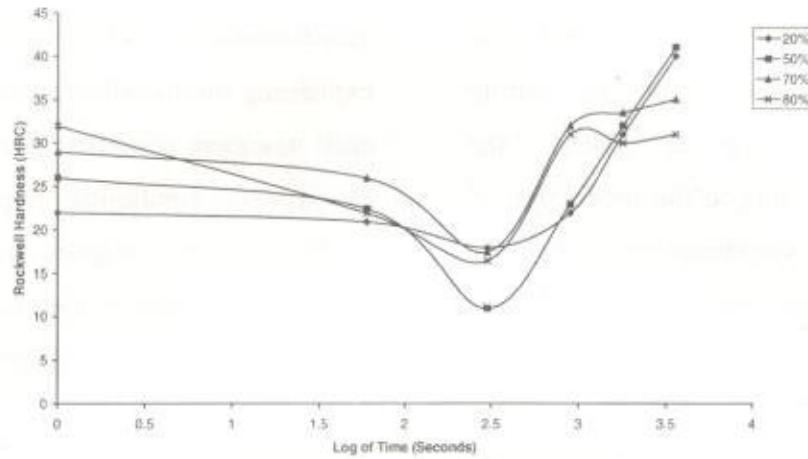


Figure 3: Rockwell Hardness Vs Annealing Time Plot For Normalized Specimens Treated at 760°C

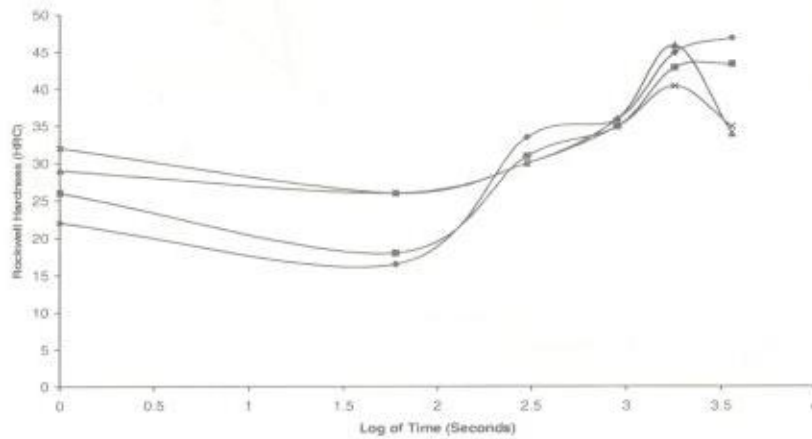


Figure 4: Rockwell Hardness Vs Annealing Time Plot For Normalized Specimens Treated at 780°C

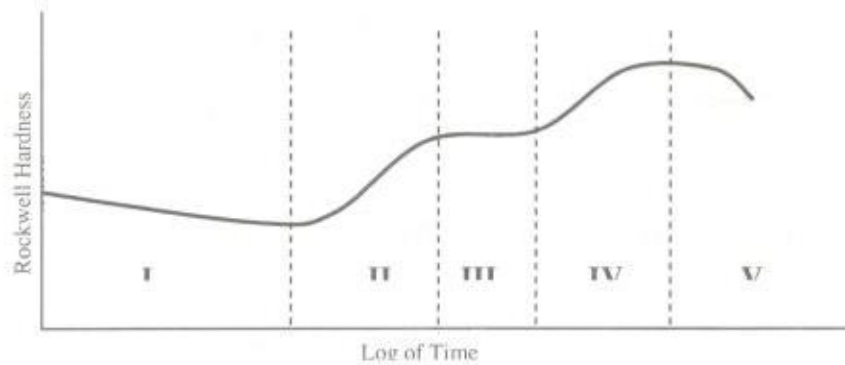


Figure 5: Schematic representation of the five stage transformation model.

Figure 6: Schematic representation of the three stage transformation model

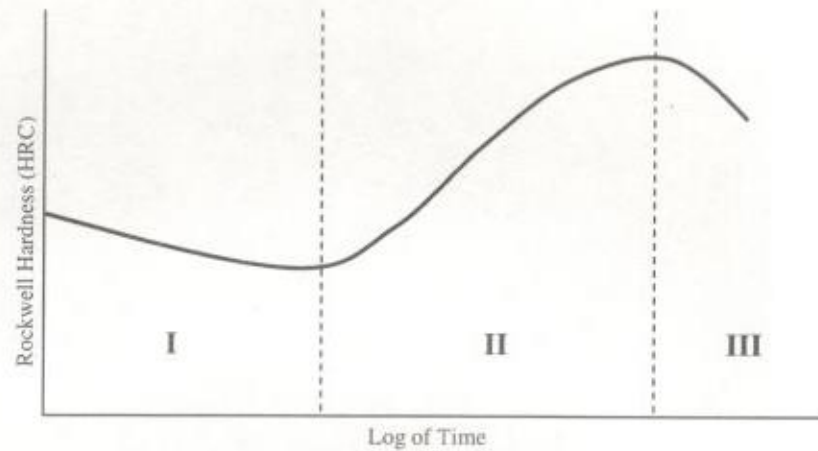


Plate 1: Photomicrograph showing the as-deformed microstructure of P/70%/ 740°C. 400X.



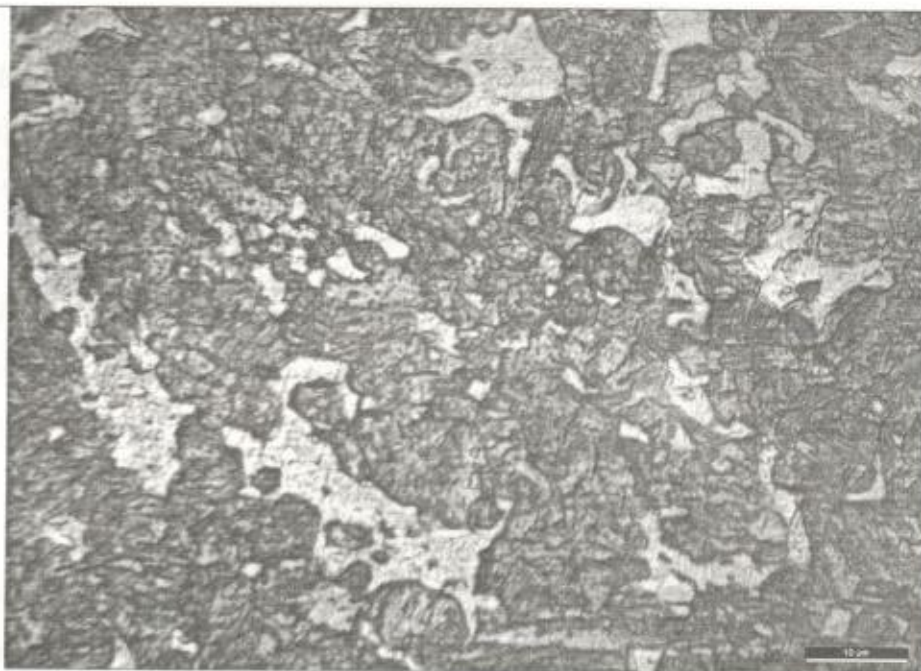
Plate 2: Showing the microstructure of P/70%/ 740°C, 1min/H₂O. The structure reveals coalesced pearlite (gray phase) and a continuous phase of ferrite (white phase) 400X.



Plate 3: Photomicrograph showing the Microstructure of P/70%/740⁰C, 15mins/H₂O. The structure reveals recrystallized grains of pearlite



Plate 4: Photomicrograph showing the Microstructure of P/70%/740⁰C, 30mins/H₂O. The structure reveals pearlite (gray coloured phase) with dispersed Islands of Martensite (dark phase).



**Plate 5: Photomicrograph showing the microstructure of P/70%/740⁰C, 1hr/H₂O.
The structure reveals a duplex structure consisting of ferrite (white phase) and
Martensite (dark phase).**

References

1. Alaneme, K. K. (2007): Evaluation of the Phase Transformation Behaviour and Mechanical properties of Duplex microstructures in carbon steel, PhD Thesis, Federal University of Technology, Akure, Pp 1 - 180.
2. Cota, A.B., Oliveira, F.L.G., Barbosa, A. L., and Lacerda, C.A.M., and Araujo, F. G. (2003): Microstructure and Mechanical Properties of a Micro-alloyed Steel After Thermal Treatments, *Materials Research*, Vol.6, No. 2, Pp 117 – 121.
3. Doherty, R.D., Hughes, D.A., Humphreys, F.J., Jonas, J., Jonsen, J. D., and Kassner, M. E. (1997): Current Issues in Recrystallization: a review, *Materials Science and Engineering A*, A238, Pp 219 – 274.
4. Gorelik, S. S. (1981): *Recrystallization in Metals and Alloys*, 2nd Edition, Mir Publishers, Moscow, 479 Pp.
5. Gorni, A. A. (2006): *Steel Forming and Heat treating Handbook*, Vol. 2, Sao Vacente, Brazil, Pp 4.
6. Kamma, C. M. and Hornbogen, E. (1984): Recrystallization Mechanism in Carbon Steels, *Canadian Met. Quarterly*, Canada, No. 2, Vol. 23, Pp 249 – 257.
7. Martin, J.W., Doherty, R.D and Cantor, B. (1997): *Stability of Microstructure in Metallic Systems* (2nd Edition). Cambridge: Cambridge University Press, UK.
8. Mazinani, M and Poole, W.J (2007): Deformation Behaviour of Martensite in a Low-Carbon Dual – Phase steel, *Advanced Materials Research*, Vols 15 – 17, pp 774 – 779.
9. Rios, P. K., Siciliano Jr., F., Sandim, H. R. Z., Plaut, R. L., and Padilha, A. F. (2005): Nucleation and Growth during Recrystallization, *Materials Research (Brazil)*, Vol. 8, No. 3, www.materialsresearch.org.
10. Stuwe, H.P, Padilha, A.F, Siciliano Jr, F. (2002): Competition between Recovery and Recrystallization, *Materials Science and Engineering A*, A233: Pp 361 – 367.
11. Torres, R. C. E., Sanchez, F. H., Gonzalez, A., Actis, F. and Herrera, R. (2002): Study of the Kinetics of the Recrystallization of cold-rolled low carbon steel, *Metall. Mater. Trans.* Vol. 32A, No.1, Pp 25 – 31.