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Phase Transformation Studies af a Low Alloy Steel in the ($\alpha + \gamma$) Phase Region

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This research work describes a thermo mechanical investigation of a low alloy steel treated in the ($\alpha + \gamma$) phase region. The aim is to develop a high tensile, ductile microstructure that could have a wide range of engineering applications. Recent advances in the area of precipitation, recrystallization mechanisms, and α / γ transformations provide strong background to this study. In a preliminary heat treatment, various microstructures (normalized and 450 °C tempered martensite structure) were produced and deformed to varying degrees. Subsequently, these microstructures were subjected to various intercritical temperatures (740 and 760 °C) for various times and a very high cooling rate. Light (optical) microscopic investigations were carried out to study the ensuing microstructures. Mechanical testing results (tensile and hardness values) were used to characterize the structures obtained. On analysis of the result, it was observed that well defined micro-duplex structures of ferritic and martensitic nature, possessing good combinations of strength and ductility were obtained.

Keywords: *thermomechanical, recrystallization mechanisms, micro-duplex structures, mechanical testing, crystallographic transformations*

1. Introduction

Phase transformations in crystalline solids are widely used in the design of microstructures or alloy development. Iron has various allotropic forms - it is this unique metallurgical property that makes it so popular for phase transformation considerations. In thermomechanical operations, for example, the effects of deformation and heating temperatures are very vital, usually, the chronological sequence in which the various phases appear matters a lot. In the thermomechanical treatment of steels, various metallurgical reactions are known to take place in different orders, thereby making the overall mechanism difficult to analyse and control[1,2].

In phase transformations involving carbon steels, it has been observed that problems of precipitation and recrystallization arise due to concurrent reactions taking place, mutually affecting each other. This problem is very prevalent during intercritical treatments, which are utilized to produce steel having a good combination of high strength and plasticity, due to the presence of the dual phases – ferrite and martensite. In this case precipitation and recrystallization processes compete with the $\alpha \rightarrow \gamma$ transformation making it difficult to establish optimum treatment conditions to achieve the best combination of ferrite and austenite, which on quenching in water will yield the best synergy of strength and plasticity[3]. Hence meaningful engineering use derivable from the intercritical treatment has not been fully harnessed because of inconclusive comprehension of the transformation behaviour in the steel especially those of carbon content above 0.2 wt. (%) C.

The present work is a preface to the attempt to develop a high strength –ductile microstructure in a low alloy steel by studying the phase transformation behaviour during treatment in the intercritical phase region. The role of the treatment parameters (initial microstructure, deformation, and temperature) will be well considered. Earlier studies on recrystallization and precipitation phenomenon in carbon steels[4] provide useful background to the test parameters.

2. Experimental Procedure

2.1. Equipment

The equipment used for this research are lathe machine, milling machine, miniature cold rolling machine, muffle furnace, power hacksaw machine, metallographic mounting press, grinding and polishing machines, rockwell hardness tester, tensile testing machine, and optical microscope with camera.

2.2. Materials

The material used for this research was a low alloy steel with chemical composition in wt. (%) as follows: C (0.4), Si (0.26), Mn (1.19), P (0.0419), S (0.0024), Cr (0.13), Ni (0.035), V(0.0017).

The material was first normalized to soften the steel for subsequent machining. The steel was austenitized at 860 °C for one hour, before cooling in air. Thereafter, the bulk sample was cut and machined to flat strips of thickness ranging from 2.5 to 10 mm.

2.3. Method

2.3.1. Production of initial microstructure

As the metallurgical pre-history of the as-supplied, specimens were unknown; the machined specimens were initially normalized at 860 °C for 1 hour and then cooled in air, so as to restore the original condition and to induce homogeneity in the structure. After normal-

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izing, different microstructures, serving as starting point-structures, were produced. The treatments were performed thus:

- Tempered Marten site™: M/450 °C, 1h/$H_2$0.
- Five test pieces were soaked for 1hour at 860 °C and then quenched in water: 860 °C, 1h/$H_2$0. The tempering of samples was carried out at 450 °C for 1hour and then air-cooled.
- Normalized (Pearlitic – feritic structure) P: 860 °C, 1h/air.

Five test pieces which were earlier produced from the initial normalization treatment were reserved for this purpose.

Additionally, three frequently occurring steel microstructures – here referred to as conventional steel microstructures were produced for the purpose of comparing properties with the structures produced after intercritical treatment. The structures produce are: normalized, martensite and 300 °C tempered martensite. These structures were produced in accordance with Alaneme[5].

2.3.2. Thermomechanical treatment

The samples of different initial microstructures were all deformed to various degrees of 20, 35, 50, 70, and 80%. After the cold deformation operation, the test pieces were subjected to intercritical heating at temperatures of 740 and 760 °C for various times, ranging from 30 seconds to 60 minutes, before quenching in water.

2.3.3. Hardness measurement

The thermomechanically treated samples were subjected to hardness testing, using a Rockwell Hardness Tester. The hardness test was used to establish the different stages of transformation. The results of Hardness (HRB) Vs logarithm (base ten) of holding time is shown in Figures 1-4.

2.3.4. Metallography

Microstructural characterization of the test specimens was performed using light miscroscopy. The specimen preparation for the light microscopy was conducted according to standard procedures[6-8]. The mechanisms of transformation (recrystallization and austenite/ferrite reaction) as well as structural features were studied.

2.3.5. Tensile testing

Mechanical property characterization was done by the study of the tensile properties of selected microstructures obtained from

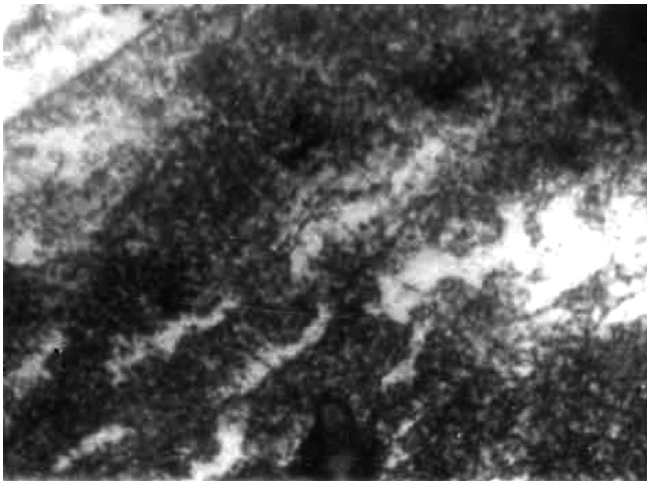

Figure 1. Photomicrograph showing the as-deformed microstructure of P/80%/760 °C. The structure reveals deformed pearlite (dark phase) and ferrite (white phase). The orientation of the ferrite indicates the direction of cold rolling. 400×.

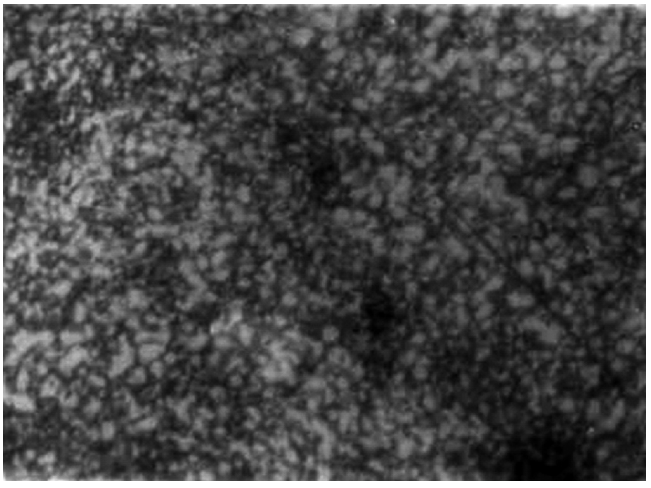

Figure 3. Photomicrograph showing the microstructure of P/80%/760 °C, 15min/H_2O. The structure reveals primary recrystallized grains of pearlite and ferrite. 400×.

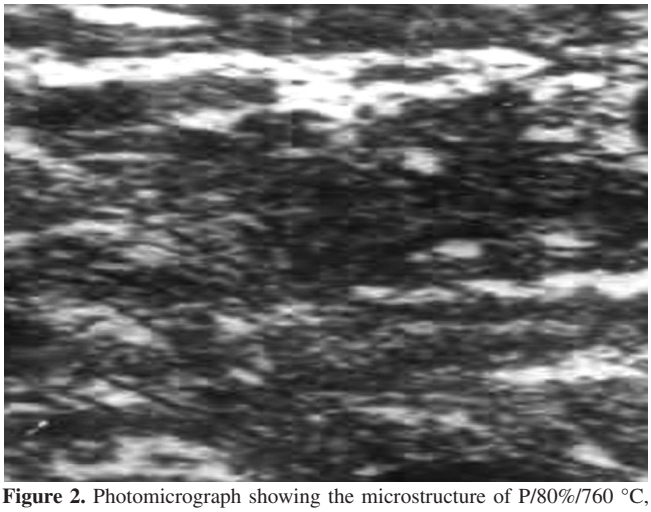

Figure 2. Photomicrograph showing the microstructure of P/80%/760 °C, 1min/H_2O. The structure reveals elongated pearlite (dark phase) and ferrite (white phase). The ferrite grains are oriented along the direction of cold rolling. 400×.

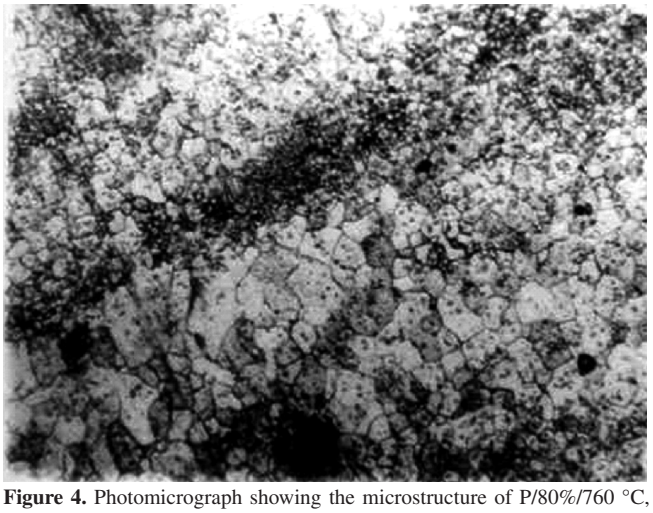

Figure 4. Photomicrograph showing the microstructure of P/80%/760 °C, 30 min/H_2O. The structure reveals well defined duplex structure of ferrite (white phase) and martensite (dark phase). The upper region shows that recrystallization is still in progress. 400×.

the intercritical treatment and also the conventional steel heat-treatment. The tensile properties evaluated were the ultimate tensile strength and percentage elongation. The samples designation and corresponding treatment for the tensile test specimens are shown in Table 1.

3. Results and Discussion

3.1. Results

3.1.1. Micrographs

Figure 1-5 are representative micrographs showing the microstructural evolution that occurs during intercritical treatment. It confirms that intercritical treatment develops micro-structures that exhibit micro-duplex features, made up of a ferritic zone combined with martensitic (formerly austenitic) zone (Figures 4 and 5).

3.1.2. Rockwell hardness

The graphs of Hardness vs. log soaking time for the various starting-point microstructures and intercritical temperatures (740 and 760 °C) are shown in Figures 6-9. The curves have the following trend: First, a gradual fall in hardness (signifying recovery processes), second a further sharp fall showing that intense spheroidization and primary recrystallizaton process are dominant at this stage; thirdly, a sharp rise in hardness signifying the formation of increased amount of martensite; and finally a decrease in hardness indicating grain growth. Thus the general transformation trend is observed to progress in four stages.

3.1.3. Tensile testing

Figures 10 and 11 show the plots of Ultimate tensile strength and percentage elongation for selected micro-duplex structures and conventional heat-treatment produced normalized, martensite, and 300 and 450 °C tempered martensite structures. The plots show that the micro-duplex structures maintain good ductility at high strength levels, in comparison with the conventional structures.

3.2. Discussion

3.2.1. Initial microstructure

The pearlite is an equilibrium structure that can scarcely undergo further phase change during the intercritical heating. Hence, on prolonged heating at the intercritical temperature region, the only possible change that can occur before commencement of $(\alpha + \gamma)$ transformation is coalescence of the cementite in the pearlite structure[9].

The tempered martensite structures consist of dispersion of ferrite and cementite.

The M450 (tempered martensite structures) is a meta-stable structure capable of further precipitation on heating at a higher temperature; though the intensity of the precipitation process will be governed by the degree of meta-stability[10].

3.2.2. Influence of intercritical treatment on microstructures

Figures 1-5 show that changes occur in the microstructure of the steel with increasing holding time during intercritical treatment. The structural evolution is brought about by the predominant transformation mechanism at the particular stage of treatment that is with respect to time. Figure 1 show the as-deformed structure, which is observed to have

Table 1. Tensile test sample designations and corresponding treatments.

Sample designation	Treatment type	Treatment routine
A	Intercritical treatment	860 °C, 1 h/H_2O + 70% deformation, 760 °C, 30 min/H_2O
B	Intercritical treatment	860 °C, 1h/H_2O + 50% deformation, 760 °C, 30min/H_2O
C	Normalizing treatment	860 °C, 1h/air
D	Quench hardening	860 °C, 1h/H_2O
E	Tempering treatment	860 °C, 1h/H_2O + 300 °C, 1 h/air
F	Tempering treatment	860 °C, 1h/H_2O + 450 °C,1h/air

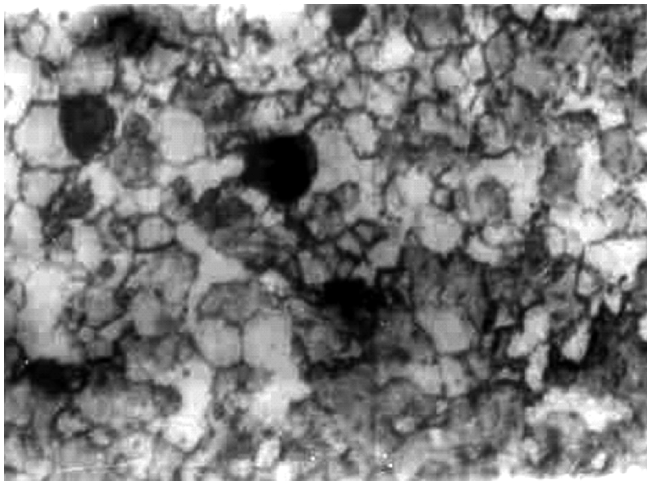

Figure 5. Photomicrograph showing the microstructure of P/80%/760 °C, 1h/H_2O. The structure reveals well defined duplex structure of ferrite (white phase) and martensite (dark phase). 400×.

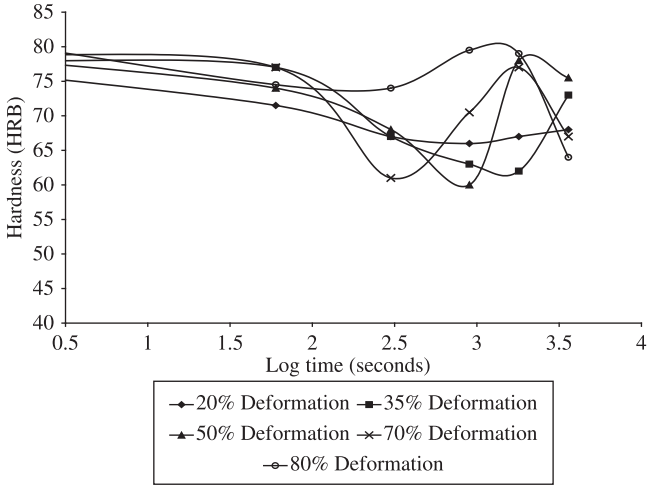

Figure 6. Variation of hardness (HRB) with annealing time for normalised specimens at 740 °C.

little difference in terms of constitution and orientation in comparison with Figure 2 – the structure after one minute holding at 760 °C before water quenching. This confirms the position that the transformation mechanism at this stage is recovery. Figure 3 shows a structure that is undergoing recrystallization, thus indicating that within fifteen minutes of treatment the transformation mechanism changes from recovery to primary recrystallization. Figure 4, which shows a duplex microstructure of ferrite and martensite, and some recrystallized and unrecrystallized zones, affirming that $\alpha \rightarrow \gamma$ transformation is preceded by primary recrystallization; while Figure 5 shows that little grain growth has occurred thus the operating mechanism is grain growth. These observations show that the chronological order of the four transformation mechanisms is: recovery → primary recrystallization → $\alpha \rightarrow \gamma$ transformation → grain growth. Figures 4 and 5 are both micro-duplex structures as they consist of the dual phase - ferrite and martensite.

3.2.3. Influence of intercritical treatment on the transformation behaviour

Figures 6-9 show the Plots of hardness (HRB) variation with annealing time at different degrees of deformation. From Figure 6, it is observed that at low deformation levels (20 and 35%) the transformation process is sluggish as the third and fourth stages of transformation are not attained. For high deformation levels (50, 70, and 80), it is observed that peak hardness (marking end of third stage) and the fourth stage of transformation was attained: indicating that transformation rate increased with increase in degree of deformation. However it is observed that for the highest deformation level (80% deformation), there is absence of second stage of transformation – little or no reduction in hardness level was observed indicating that primary recrystallization process was suppressed in favour of continued recovery (polygonization).

For the normalized samples treated at 760 °C (Figure 7), it is observed that for all deformation levels, the four stages of transformation was attained. This indicates that the transformation is completed within 60 minutes when a higher temperature of 760 °C is utilized. Thus indicating that utilizing a higher temperature increases the transformation rate. However, it is observed that for the 80% deformed sample the onset of the third stage was delayed. This indicates that extremely high deformation levels (≥ 80%) can slow down the transformation rate. Kamma[1] has attributed this to be due to the broadening of the deformation bands and homogenization of the structure leading to retardation of the transformation process.

The M450 tempered martensite specimens exhibited slower transformation rates at 740 °C (Figure 8) in comparison to the normalized specimens (Figure 6). This suggests that the normalized initial microstructure averagely yields faster transformation rates in comparison to the M450 specimens. This can be attributed to the presence of Fe_3C particles, which during treatment undergo further precipitation and growth, thereby slowing down or pinning the transformation fronts formed by the nucleating γ grains. For the M450 specimens treated at 760 °C (Figure 9), it is observed that the onset of the third stage of transformation is longer for the heavily deformed specimens (70 and 80% deformation levels) in comparison with the utilization of lower deformation levels. This indicates that increasing deformation levels does not necessarily favour increased transformation rate and can even bring about retardation of the process. But on comparison with Figure 8 – M450 specimens treated at 740 °C - it is observed that on the average that transformation rate is faster at 760 °C than at 740 °C, which is in agreement with Yang and Chen[11] and Cota et al.[12].

3.3. Comparison of tensile properties of micro-duplex structures with conventional structures.

Figures 10 and 11 show the tensile properties of selected micro-duplex structures (P/70%/760 °C, 30 min/H_2O and P/50%/760 °C, 30 min/H_2O – with microstructures obtained at the peak of the third stage of transformation); which were used as test case to compare tensile properties with

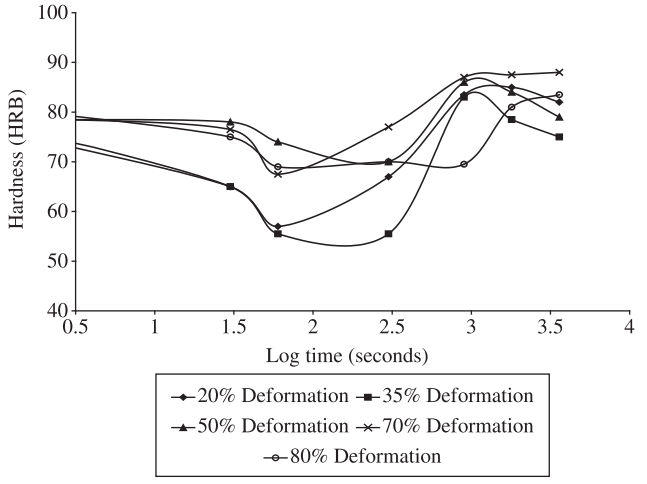

Figure 7. Variation of Hardness (HRB) with Annealing Time for Normalised Specimens at 760 °C.

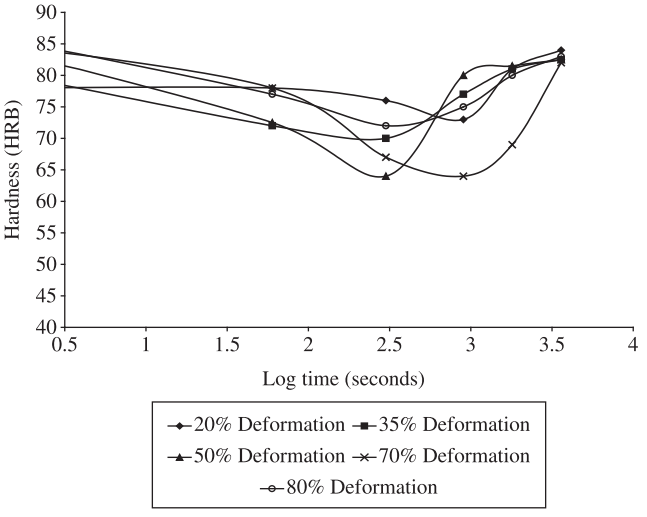

Figure 8. Variation of hardness (HRB) with annealing time for 450 °C tempered martensite specimens treated at 740 °C.

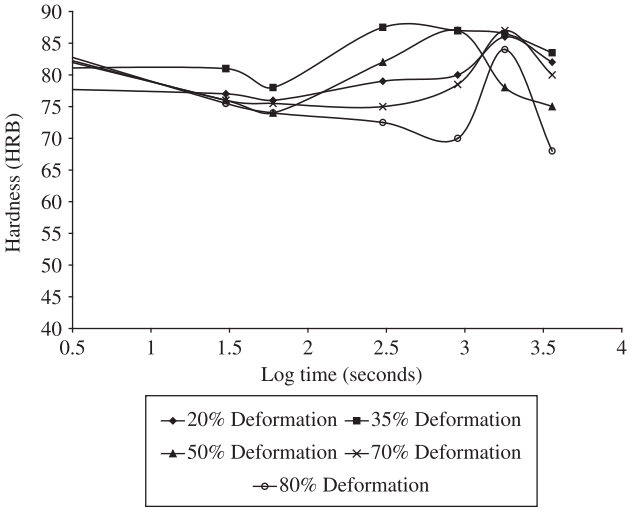

Figure 9. Variation of hardness (HRB) with annealing time for 450 °C tempered martensite specimens treated at 740 °C.

that of conventional heat-treated normalized, martensite and tempered martensite structures (M300 & M450). From Figure 10 it is observed that the micro-duplex structures (A and B) had tensile strength values of 878 and 870 N.mm^{-2}, which were higher than that of the conventional heat-treated structures (C – normalized, D -martensite, E – 300 °C tempered martensite structure and F – 450 °C tempered martensite structure). This indicates that when optimum intercritical treatment conditions are selected improved tensile strength could be obtained[13,14]. The advantage of the process is underlined when the percentage elongation values are compared (Figure 11). From the chart it is observed that the micro-duplex structures (A and B) equally have the highest percentage elongations of 24 and 25% respectively. These values in comparison with those of the conventional heat-treated specimens show that optimum structures obtained from the intercritical treatment have very good ductility at high strength levels - thus the treatment yields improved plasticity and toughness in the low alloy steel while maintaining high strength. The tensile strength and percentage elongation values obtained from the intercritical treatment is within the range reported by Podder et al.[15] and Speer and Matlock[16]. This indicates that properly determined treatment parameters can help yield duplex microstructures of ferrite and martensite of the right volume proportion that will yield a good combination of tensile strength and ductility.

4. Conclusion

Phase transformation studies of a low alloy steel in the intercritical phase region has been investigated. The aim being to develop high strength – ductile microstructures in the low alloy steel. From the results obtained the following conclusions are drawn:

Phase transformation during intercritical treatment of the low alloy steel progresses in four transformation stages; and in the order: recovery → primary recrystallization → α / γ transformation → grain growth.

Transformation rates are faster when 50 and 70% deformations prior to intercritical treatment are utilized than when lower deformation levels (20 and 35%) and very heavy deformations (≥ 80%) are utilized; also transformation rates are faster at 760 °C than at 740 °C.

Intercritical treatment performed by selecting the normalized initial microstructures, deformed to 50 and 70% before treating at 760 °C for 30 minutes and then water quenched; yielded micro-duplex structures combining high strength and good ductility superior to that of conventional heat-treatment steel structures.

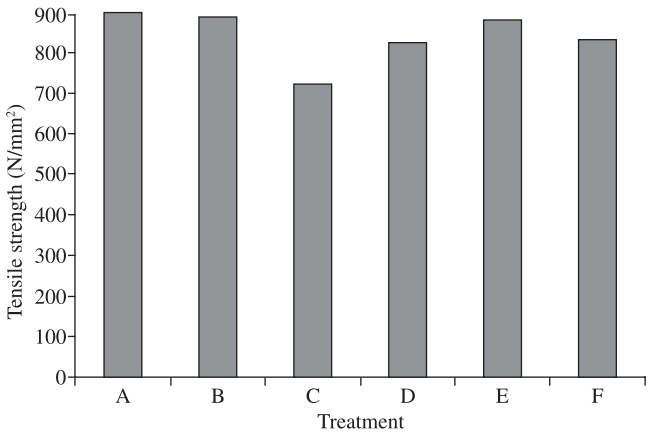

Figure 10. Comparison of tensile strength of selected micro-duplex structures with that of conventional structures.

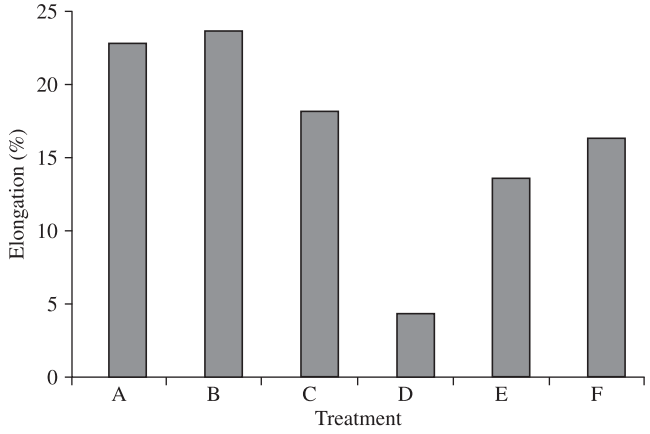

Figure 11. Comparison of percentage elongation of selected micro-duplex structures with that of conventional structures.

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