Microstructure Evolution and its Influence on Tensile Behavior of Process Annealed Cold Drawn 0.12wt% C Steel

Nurudeen A. Raji, Oluleke O. Oluwole

Abstract- This work studied the effect of grain size evolution of cold drawn 0.12wt% C steel subjected to process annealing on tensile behavior. 20%, 25%, 40% and 55% cold drawn 0.12wt% C steel were subjected to annealing comprising of slow heating-up to various temperature ranging from 500°C to 700°C at interval of 50°C followed by soaking treatment for 10 minutes, 20 minutes, 30 minutes, 40 minutes, 50 minutes, 60 minutes under each of the temperature in a muffle furnace. These samples were submitted to optical microscopy analysis and to tensile test. After annealing at 650°C and soaked for 10 minutes, the dislocation defects were annihilated in the 25% cold drawn samples. Grain coarsening is observed for the annealed steel at soaking time of 20 minutes to 30 minutes after which grain growth commenced at annealing temperature above 650°C at soaking time of 40 minutes for the 25%, 40% and 55% cold drawn samples. Fine grains of the microstructure were observed for all the annealed samples between the temperature range 500°C-650°C. The yield strength of the annealed samples increases compared to the non-treated samples thus improving the ductility of the steel. A better improvement of the yield strength is observed for the annealing temperature of 500°C and 550°C at soaking time of 10 minutes and 30 minutes for all the cold drawn samples except for the annealed 25% cold drawn steel whose yield strength is below the yield strength of the non-treated samples.

Index Terms— annealing, cold drawn, deformation, grain size, microstructure, tensile strength, yield strength, steel

1 INTRODUCTION

The properties of polycrystalline materials depend on the microstructure and the properties of single crystal. From the viewpoint of microstructure, many factors contribute to the macroscopic properties. Grain size distribution, grain boundary and misorientation distribution are important to the mechanical properties of the material [1].

When a metal is cold drawn, the microstructure presents a morphological texture where the grains are lengthened along the wire drawing axis [2], [3]. The wire hardens during plastic deformation and the ductility is reduced while the tensile strength increases. The structural hardening is due to the movement of dislocation and the generation of additional dislocation within the material structure. This defect is known as strain hardening and is usually accompanied with reduced ductility of the material [4]. The distorted, dislocated structure resulting from cold working of the metal becomes unstable due to the strain hardening effect and a heat treatment procedure known as annealing is usually used to modify these defects and improve the mechanical properties of the material [5]. Annealing is a heat treatment procedure wherein a material is altered causing changes in its properties such as strength and hardness. It is used to induce ductility, soften material, relieve internal stresses, and refine the structure by making it homogenous, and improved cold working properties. The modifications of the material during annealing occur in three stages known as recrystallization processes, according to temperature or time. These are; recovery, recrystallization and grain growth.

The microstructure evolution of materials can be studied using various techniques which include the X-ray diffraction (XRD) analysis [6], [7], transmission electron microscopy analysis (TEM) [8], [9], [10], [11] and electron backscattered pattern analysis (EBSP) [12], [13], [14], [15]. Different empirical, phenomenological, and first-principle models exist to predict the behavior of these microstructure phenomena over a variety of conditions. These methods have been successfully used to determine the crystal structure, phase transformation temperatures, and precipitation behavior of annealed specimen. It is also possible to non-destructively measure the growth kinetics of individual bulk grains in-situ by 3-dimensional X-ray diffraction (3DXRD) microscopy [16], [17]. Three-dimensional X-ray diffraction (3DXRD) allows non-destructive investigation of the microstructures in the bulk of crystalline materials [18]. 3DXRD has been used by a multitude of users for a range of different applications.

• Nurudeen A. Raji is currently pursuing PhD degree program in Mechanical engineering in University of Ibadan, Nigeria. E-mail: kunle_raji@yahoo.com

• Dr. Oluleke O. Oluwole is a Senior Lecturer in the Mechanical engineering department of the University of Ibadan, Nigeria. E-mail: oluwoleo2@asme.org
including recovery [19], nucleation and growth during recrystallization [16], [20], elastic strains in individual bulk grains [21], phase transformations [22] and sub-grain dynamics during plastic deformation [23]. The nucleation and growth of new grains during recrystallization of deformed metals can be followed in situ by 3DXRD [24].

The EBSP method offers the advantage of being able to study the effect of impingement between recrystallizing grains directly. The EBSP also allow the direct investigation of the effects of boundary misorientation on boundary migration rates, because the data contain a record of the orientation of pixels in the deformed matrix which are consumed by each specific recrystallizing grain during any given annealing step.

It is evident from all the various studies that microstructural evolution of materials, during annealing, may involve a wide variety of phenomena, occurring simultaneously or sequentially [25], [26]; grain shape change due to material flow, work hardening, dynamic recovery, dynamic recrystallization, metadynamic recrystallization, static recrystallization, static recovery, phase transformation, precipitation, and grain growth. The kinetics of these thermally activated phenomena are strongly dependent on the thermal profile as characterized by heating rate, annealing temperature, soaking time, and cooling rate. These heating parameters control the microstructural features such as grain size distribution, shape, and texture, which in turn determine the mechanical properties (such as strength, hardness, and ductility) that are crucial for the end applications[27], [28].

The recrystallization kinetics could be used to describe the microstructure evolution of metals subjected to annealing heat treatment. The annealing parameters such as the annealing temperature, soaking time, and heating rate has various influence on the microstructural features, such as grain size distribution, grain shape anisotropy, and grain orientation of the metal [29]. This influence of annealing parameters on the microstructure has been demonstrated for the microstructural size and shape parameters during annealing of cold rolled aluminum killed steel strips examined under non-isothermal condition [30]. It was shown that decrease in the heating rate results in accelerated grain growth behavior.

This paper focuses on the microstructure evolution, the tensile properties, impact toughness and the average grain size of the annealed cold drawn low carbon steel as applicable for the manufacture of plain nails. With the knowledge of the grain size, it is possible to predict the response of the steel to heat treatment and behavior during working or under various stress conditions to which it may be subjected during service.

2 EXPERIMENTAL PROCEDURE

The material studied in this work was a commercially available low carbon wire steel rod with the composition in wt. % as shown in Table 1. The steel was cold drawn in a series of drawing dies reducing the wire diameter from 5.5 mm to 5 mm, 4 mm, 3.3 mm, and 3 mm representing respectively 20%, 25%, 40% and 50% degree of deformation as is applicable for the manufacture of the 4 inches, 3 inches, 2½ inches and 2 inches plain nails respectively. Subsequently samples of the wires cut to 50 mm lengths were annealed in a Muffle furnace at 900 deg. C for 10 min, 20 min, 30 min, 40 min, 50 min, and 60 min soaking time and cooled in the furnace to ambient temperature of 27 deg. C.

| Table 1 CHEMICAL COMPOSITION OF THE AS-RECEIVED LCS WIRE MATERIAL (WT.) [32] |
|-----------------|---|---|---|---|---|
| C               | Si | Mn | P  | Fe |
| 0.12            | 0.18 | 0.14 | 0.7 | 98.86 |

The metallographic specimens were sliced from each of the annealed cold drawn steel. The samples for evaluation of the microstructures by optical microscopy (OM) were cut from the annealed wire, and taken through a grinding process on silicon carbide paper, 240, 320, 400, and 600 grit. The samples were then polished initially at 1μm and finally at 0.5μm using emery cloth and silicon carbide solution, etched with 2% nital and observed under the optical microscope (OM) with image capturing device. The average grain sizes of the specimens were obtained using the grain counting procedure [31].

The tensile tests were conducted at ambient temperature of 27 deg. Con a universal testing Instron 3369 with a load capacity of 50kN to obtain the strength-ductility properties of the samples at the different soaking time. The gauge dimension of each specimen for the tensile test is 45mm length. Three samples each of the annealed steel were employed to obtain set of mechanical data. The yield strength, tensile strength and ductility of the specimens were determined as obtained in [32].
3 RESULTS AND DISCUSSION

3.1 Microstructure Characterization
The microstructure evolution during recrystallization was characterized in terms of the grain size. The sub-grain size at the initial stage of recrystallization for the 20%, 25%, 40% and 55% degree of cold drawn deformation annealed at temperature range 500°C-700°C is shown in Fig. 1. It was observed that the sub-grain size of the annealed 25%, 40% and 55% cold drawn 0.12wt% C steel decreased with increasing degree of deformation. However the sub-grain formation of the annealed 20% cold drawn steel showed a contrary response. This implies that increasing degree of cold drawing introduced smaller sub-grains thereby increasing the number of grains nucleating site for recrystallization. The sub-grain size increased with increasing annealing temperature as shown in Fig. 2. This implies that the starting sub-grain size for recrystallization depends on annealing temperature suggesting that the rate of recrystallization increases with increase in annealing temperature. It is therefore expected that complete recrystallization would occur according to the annealing temperature.

Fig. 1. Sub-grain size distribution chart

Fig. 2. Influence of annealing temperature on sub-grain size

3.2 Mechanical Properties
Fig. 3-10 shows the properties dependence on the soaking time of annealing of the cold drawn steel for the 20%, 25%, 40% and 55% degree of deformation annealed at temperature within the range of 500°C-650°C. The yield strength of the annealed samples improved when compared with the as-received control sample (CS) of the
steel for all the degrees of cold drawn steel. A better improvement of the yield strength is observed for the annealing temperature of 500°C and 550°C between the soaking time of 10 minutes and 130 minutes after which the rate at which the yield strength increases for the treated samples reduces with increasing temperature of annealing for all the degrees of cold drawn deformation. Improved yield strength is also recorded for the 40% degree cold drawn steel at 650°C compared to when annealed at 500°C at soaking time above 30 minutes. The 25% cold drawn steel annealed at 650°C have lower yield strength compared to the yield strength of the control sample.

The tensile strength of the annealed samples reduced considerable for all the degrees of cold drawn steel annealed between 500°C and 650°C except for the 20% degree of cold drawing where the strength improved by 2.3% and 0.8% between soaking time of 10 minutes and 20 minutes respectively when annealed at 500°C.

Fig. 3. Yield strength response of annealed 20% cold drawn 0.12wt% C.

Fig. 4. Tensile strength response of annealed 20% cold drawn 0.12wt% C.
Fig. 5. Yield strength response of annealed 25% cold drawn 0.12wt% C.

Fig. 6. Tensile strength response of annealed 25% cold drawn 0.12wt% C.

Fig. 7. Yield strength response of annealed 40% cold drawn 0.12wt% C.
Fig. 8. Tensile strength response of annealed 40% cold drawn 0.12wt% C.

Fig. 9. Yield strength response of annealed 55% cold drawn 0.12wt% C.

Fig. 10. Tensile strength response of annealed 55% cold drawn 0.12wt% C.
3.4 Influence of Annealing Temperature on Grain Size

The influence of the annealing temperature on the grain size of the annealed cold drawn steel samples is shown in Fig. 13-16. Finer grain sizes were observed for all the soaking time steel samples annealed at temperature range between 500°C and 650°C for the 20% cold drawn steel as shown in Fig. 13. The grain size increased slowly with increasing annealing temperature up to 650°C after which further annealing at temperature above 650°C results in rapid grain growth. Similar trend was observed for the annealed 25%, 40% and 55% cold drawn steel between annealing temperature of 500°C to 600°C as shown in Fig. 14-15. It could be explained that grain growth of recrystallized grains commenced after the upper limits of these temperature range. It is therefore clearly evident that grain coarsening in the annealed commenced after 650°C for the 20% cold drawn deformation and 600°C for the 25%, 40% and 55% cold drawn deformation.

Fig. 13. Influence of annealing temperature on grain size of 20% cold drawn 0.12wt%C steel

Fig. 14. Influence of annealing temperature on grain size of 25% cold drawn 0.12wt%C steel
Fig. 15. Influence of annealing temperature on grain size of 40% cold drawn 0.12wt%C steel

Fig. 16. Influence of annealing temperature on grain size of 55% cold drawn 0.12wt%C steel

3.5 Influence of Soaking Time of Annealing on Grain Size

The influence of the soaking time on the grain size as shown in Fig. 17-20 reveals that the supposed finer grain formation also depends on the soaking time. Fig. 17 and 18 shows that for the annealed 20% and 25% cold drawn deformation, the grain size increases with increasing temperature and finer grain size could be obtained between 500°C and 550°C and soaking time range of 10 minutes to 30 minutes. Similar observation for the 40% degree of cold drawn deformation show that finer grain size could be obtained between 500°C and 600°C for soaking time range of 10 minutes to 30 minutes and also between 500°C and 550°C within soaking time of 10 minutes and 30 minutes for the 55% degree of cold drawn deformation.
Fig. 17. Influence of soaking time on annealed 20% cold drawn 0.12wt%C steel

Fig. 18. Influence of soaking time on annealed 25% cold drawn 0.12wt%C steel
4CONCLUSIONS

The influence of the annealing heat treatment on the microstructure and tensile strength and yield strength properties of cold drawn 0.12wt% C steel was analyzed using the optical microscopy and tensile test experiment. The grains of the microstructure elongate as the steel is cold drawn. The elongation of the grains increases as the degree of cold drawn deformation increasing. The microstructure is found to contain non-uniformly concentrated dislocation defects at higher degree of cold drawn deformation of 40% and 55%. After annealing at 650°C and soaked for 10 minutes, the dislocation defects were annihilated in the 25% cold drawn samples. Grain coarsening is observed for the annealed steel at soaking time of 20 minutes to 30 minutes after which grain growth commenced at annealing temperature above 650°C at soaking time of 40 minutes for the 25%, 40% and 55% cold drawn samples.

Fine grains of the microstructure were observed for all the annealed samples between the temperature range 500°C-650°C. The grain size increases slowly with increasing annealing temperature between 500°C and 650°C for soaking time range of 10 minutes to 30 minutes.
The grain size evolution of the microstructure has considerable influence on the tensile properties of the samples. The yield strength of the annealed samples increases compared to the non-treated samples thus improving the ductility of the steel. A better improvement of the yield strength is observed for the annealing temperature of 500°C and 550°C at soaking time of 10 minutes and 30 minutes for all the cold drawn samples except for the annealed 25% cold drawn steel whose yield strength is below the yield strength of the non-treated samples.

REFERENCES


