

ORIGINAL ARTICLE

Effect of Cold Rolling and Flash Annealing on Microstructure and Mechanical Behaviour of Austenitic Manganese Steel (Fe-1.15C-13Mn)

A. H. Al Hakam¹, F. M. Ridlo^{2,*}, R. A. Tanjung¹, L. A. Damar¹ and P. A. Paristiawan²¹Department of Earth and Environmental Sciences, Materials and Metallurgical Engineering Study Program, Institut Teknologi Kalimantan, Balikpapan, Indonesia²Research Center for Metallurgy, National Research and Innovation Agency (BRIN), Tangerang Selatan, Indonesia

ABSTRACT – Austenitic manganese steel (AMS) is widely used in the mineral and mining industry under high workload condition due to its remarkable work-hardening capacity under impact. However, AMS typically has an austenitic matrix with carbides precipitated resulting in low hardness and brittle properties, which impaired its wear performance. In this study, cold deformation followed by flash annealing were conducted to improve the mechanical behavior of AMS. The experimental was carried out by solution treatment (ST), cold rolling (CR) with deformation degrees of 10% and 20%, and flash annealing (FA) at 915°C with holding times of 90 and 150 seconds. The microstructure evolution and mechanical behavior were studied. The ST produced a completely austenitic microstructure which was free of carbide, resulting in a decrease in strength and hardness, as well as improved ductility. After cold deformation, both of strength and hardness were substantially improved, followed by loss in ductility. Optical microstructures reveal the formation of deformation twin (DT) and annealing twin (AT) after cold deformed. Higher intensity of DT results in AMS with considerable strength and hardness but decrease in ductility. After FA process, cold deformed structures undergo microstructural restoration, which manifests by recovery stage at 90 s and recrystallisation stage at 150 s annealing time. At the same FA time, higher degree of deformation led to increase the hardness, while at the same degree of strain, longer annealing time led to a decrease of hardness. Moreover, both of higher degree of strain and longer annealing time during flash annealing were contributed to grain refinement, even though did not affected to the increase of tensile and hardness. In addition, 20% cold rolling followed by 150 s annealing time (CR20FA150) could be considered as an effective method to obtain the most optimum combination of strength and ductility with finer grain.

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INTRODUCTION

Austenitic manganese steel (AMS) is a type of steel which is frequently used in heavy industrial applications, such as mining equipment, mineral processing industries, railroads [1], and military applications (special helmets and security armor lining-plates [2]. AMS, also known as Hadfield steel, has a carbon to manganese ratio of 1:10 and a chemical composition of 1–1.4% C and 11–14% Mn, resulting in an austenitic microstructure which is stable at room temperature [3]–[5]. It is very common that in the as-cast state, AMS contains some carbides precipitated at the grain boundaries [6], typically (FeMn)₃C carbides [7]. Therefore, solid solution treatment is one type of heat treatment method that is commonly used to generate homogeneous austenitic structure in AMS [8]–[9].

Considering the austenitic matrix at room temperature, AMS typically does not have high enough hardness [6], but it is characterized by high strain hardening capacity with reasonable ductility. Some previous studies have revealed that possible hardening mechanism for AMS is either by adding other chemical elements (Cr, Ni, V, Ti or Nb) [10]–[14] or by cold deformation [15].

The work hardening caused by deformation will increase hardness value to a certain depth and significantly affects the wear performance of the AMS. However, in practice, due to low hardness, AMS is not able to get sufficient work hardening, which limits the application of these steel. This material, in as-quenched condition, has a hardness value of 200 BHN and can increase up to 600 BHN due to its work hardening abilities [14], [16]–[17]. Furthermore, the work hardening mechanism is determined by formation of high dislocation density as well as mechanical twinning, which results in enhancement of strength due to interactions between dislocations and twins [18].

However, the strength improvement by cold deformation exhibits lowering ductility to AMS. Therefore, the subsequent annealing treatment will be required to recover the mechanical properties. This research is aimed to get a further understanding of the deformation mechanism by identifying the correlation between microstructure evolution and mechanical behavior of AMS after subjected by cold deformation and flash annealing process. In this paper, the effect of strain degree and annealing time on the structure-properties of AMS is elaborated.

EXPERIMENTAL METHOD

Materials and Instruments

The austenitic manganese steel (AMS) used in this experiment is commercial CREUSABRO mangan supplied by PT. Tyra austenite with the chemical composition listed in Table 1.

Table 1. Chemical composition of commercial CREUSABRO

Element	C	Mn	Si	S	P	Fe
Wt.%	1.15	13	0.4	0.03	≤0.045	Bal.

The solution treatment and flash annealing process were performed using Nabertherm muffle furnace LH-15/14"/C440 series. Cold deformation was conducted with two high-mill rolling machine, with 10% and 20% deformation degree. The mechanical tests including hardness testing using BREVETTI AFFRI with Brinell method using 2.5 mm diameter steel ball indenter and 187.5 kg load according to ASTM E10, while room temperature quasi-static tensile testing using Tinius Olsen machine with subsize specimen according to ASTM E8M. The microstructures evolution were analyzed using light optical microscope Olympus BX53M equipped with grain size intercept software for grain size measurement.

Method and Procedure

Prior to heat treatment process, the AMS samples were prepared with dimensions of 180 × 15 × 6 mm, as shown in Table 2.

Table 2. Classification of samples before treatment

Samples classification	Treatment		
	ST*	CR* (%)	FA* (s)
AMS	-	-	-
AMS-ST	✓	-	-
CR10	✓	10	-
CR20	✓	20	-
CR10FA90	✓	10	90
CR10FA150	✓	10	150
CR20FA90	✓	20	90
CR20FA150	✓	20	150

*ST Solution treatment *CR Cold rolling *FA Flash annealing

The schematic of heat treatment and cold deformation process was shown in Figure 1. The solution treatment (ST) procedure involves two-steps heating method. At the first step, samples were heated from room temperature to 700°C and held for 1 hour. The heating was continued up to 1000°C, held for 30 minutes, followed by rapid cooling into agitated water.

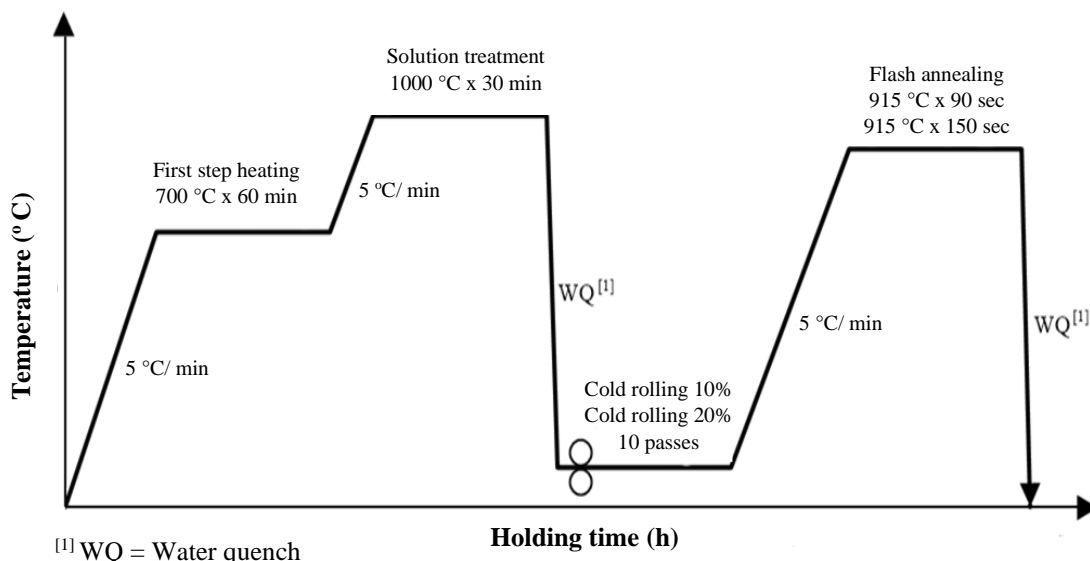


Figure 1. The schematic of solution heat treatment and cold deformation

After solution treatment process, according to Table 2, some of the samples were subjected to cold rolling process with 10% and 20% of reduction. Following cold deformation step, the flash annealing process was performed at temperature of 915°C with annealing time of 90 seconds and 150 seconds, respectively. All heat treatments were conducted with heating rate of 5°C/minutes. The mechanical tests were performed and prior to microstructure observation, several mechanical preparation steps were carried out including grinding up to grit #1500 SiC paper, polishing with 1 micron alumina, and etching with 4% Nital for 5–10 seconds.

RESULTS AND DISCUSSION

The microstructure of austenitic manganese steel (AMS) before solution treatment is demonstrated in Figure 2(a). In general, AMS has a homogeneous austenite microstructure with some annealing twin (AT) and carbide which probably appear during the hot rolling process in industrial manufacture. This is confirmed by the results of Tewary et al. [15], which explained that formation of AT presents in the microstructure of high-manganese steels subjected by hot deformation. Meanwhile, the presence of carbide is influenced by the carbide-former elements contained in the material [19], such as carbon and manganese. A large amount of carbide formation can be induced by AMS containing more than 1.2% carbon and more than 13% manganese, according to Subramanyam [20].

Figure 2(b), on the other hand, depicts the microstructure of AMS following solution treatment (AMS-ST sample) which exhibits a single-phase austenite structure with some annealing twin. However, the presence of carbides does not show. The disappearance of carbides indicates that the solution treatment process may completely dissolve the carbides into austenite. Moreover, this process can also eliminate the heterogeneity of the microstructure as indicated that the density of the annealing twin (AT) is decreased.

Figures 3 (a) and (b) show the microstructure of the AMS samples after cold rolling of 10% (CR10) and 20% (CR20), respectively. It is noticed that non-uniform formation of annealing twin (AT) and deformation twin (DT) with different intensity were observed. The twinning intensity appears to be relatively high in some grains while less intense in other grains. However, this does not imply that the grains were not plastically deformed, where the DT is not visible, as the deformation traces within the grains may be perpendicular to the viewing surface [21]. Grains with higher intensity of DT appear darker than the lower one, which appear lighter. This is due to the difference in etching speed which explains that grains with higher DT intensity tend to have a more exposed surface, causing atoms in the deformation band become more reactive during etching and result a darker area [21]. While the CR10 sample contains a few grains with darker colors and varying grain sizes, the CR20 sample appears to contain more grains with darker colors and more uniform sizes. This demonstrates that the intensity of DT increases with increasing degree of strain.

Figures 4 (a)–(d) show the microstructure of the cold rolled AMS material after flash annealing (FA) process. In general, it is clear that the microstructure consists of annealing twin (AT) and deformation twin (DT) which exhibit a significant reduction of deformation twin intensity. Therefore, it indicates that flash annealing triggers the initial stages of microstructural restoration which are characterized by the transformation of deformed grains into recovered grains. The recovered grains is demonstrated by decreasing the intensity of DT within the grain, even though some DT still exist. However, the deformation twin which still exists after flash annealing at 915°C indicates their thermal stability during the annealing process [22]. The following stage of the microstructural restoration phenomena took place at 150 seconds FA, which characterized by the formation of partial recrystallization grains, as illustrated in Figures 4(b) and (d). Raabe [23] states that this stage is defined by the nucleation of new grains (partial recrystallized grains) that develop along the parent grain boundaries and typically exhibit equiaxed shape. Besides the presence of deformation twin, the recrystallization stage during the FA process also resulted in the presence of annealing twin (AT) which is in agreement with Tucho, et.al. [24]. Moreover, according to Jin et al. [25], it was confirmed that the previous applied cold deformation affects AT formation during the recrystallization process.

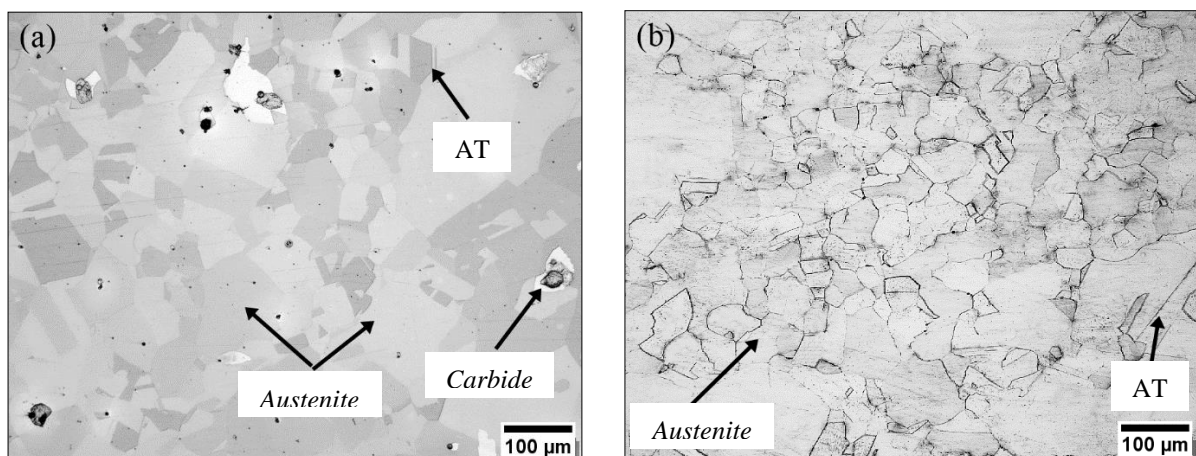


Figure 2. Optical micrograph of (a) Commercial Creusabro (AMS) and (b) AMS-ST

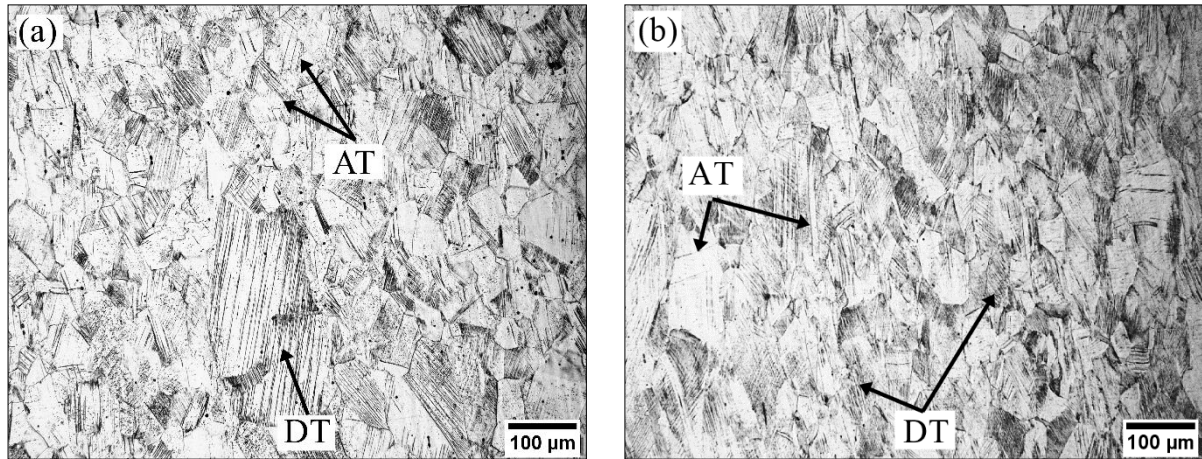


Figure 3. Optical micrograph of AMS after cold rolling (a) 10% (CR10) and (b) 20% (CR20)

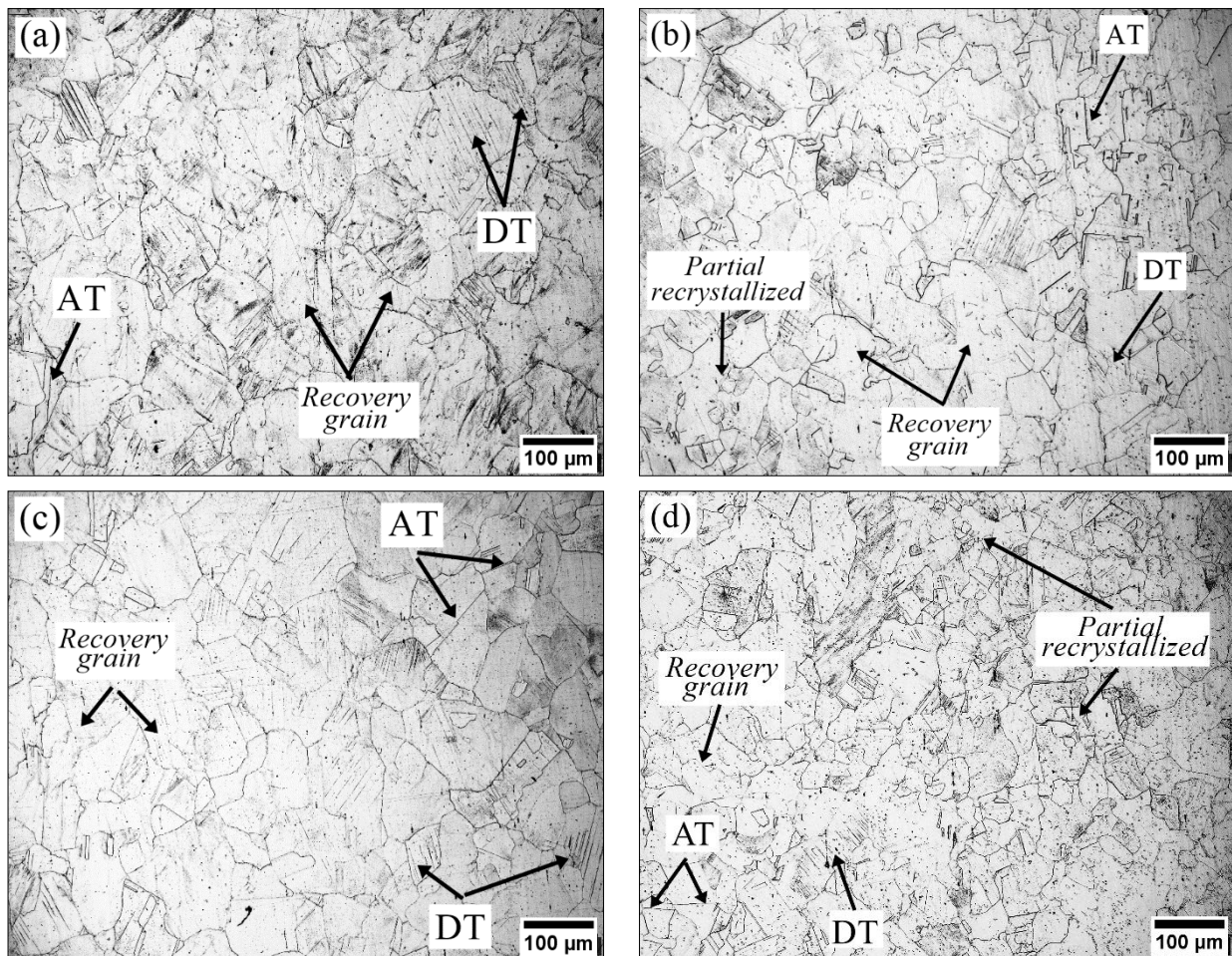


Figure 4. Optical micrograph of cold rolled AMS after flash annealing at 915°C: (a) CR10FA90, (b) CR10FA150, (c) CR20FA90, and (d) CR20FA150.

Figure 5 and Table 3 summarize the mechanical behavior, tensile test, and grain size measurement of AMS in different treatment. Both of yield strength and tensile strength of the AMS samples dropped after the solution treatment, from 380 MPa to 365 MPa and 940 MPa to 907 MPa, respectively. Even though, this strength decrement is followed by an increase in ductility, up to 52%. Microstructure analysis revealed that it was driven on by carbide dispersion into the austenite matrix, which led to a reduction in strength and an increase in plasticity. This demonstrates that parameters used during solution treatment, including temperature, holding, time and coolant media, were sufficiently effective to dissolve carbides into austenite matrix.

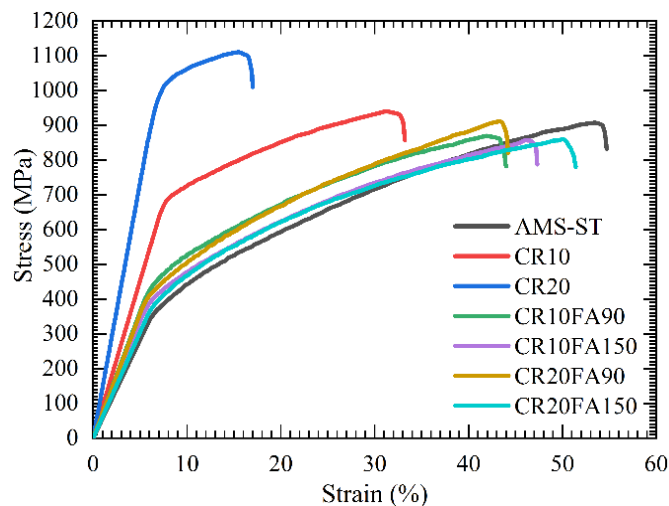


Figure 5. Engineering stress-strain of AMS in different treatment

Table 3. Tensile test results and grain size measurement of different samples

Samples code	Grain size (μm)	Yield strength (MPa)	Tensile strength (MPa)	elongation (%)
AMS	46.96	380	940	40.00
AMS-ST	38.52	365	907	52.32
CR10	32.39	678	940	28.78
CR20	27.25	965	1110	20.34
CR10FA90	60.19	429	869	37.14
CR10FA150	41.25	388	857	41.43
CR20FA90	52.61	406	911	40.86
CR20FA150	36.85	383	860	45.71

Furthermore, the cold deformation process subjected to AMS will significantly increase the yield and tensile strength. At CR10 sample, the increase in yield and tensile strength at CR10 was 678 MPa and 940 MPa, respectively. Meanwhile, at CR20 sample, which has a yield strength of 965 MPa and a tensile strength of 1110 MPa, exhibits the highest increase in strength. The strength increase was confirmed through microstructure observations indicating the presence of DT which contributes to strengthening mechanism. It is completely controlled by formation of mechanical twinning which is the main obstacles of dislocation gliding [15]. During the cold deformation process, a large number of high-density deformation twins appear, resulting in the interaction between two DT and DT with dislocations. It makes dislocation movement more difficult and triggers metal strengthening and work hardening capacity [26]. AMS is typically face-centered cubic (FCC) metal which has a low stacking fault energy (SFE) characteristic of $23\sim 50 \text{ mJ m}^{-2}$, allowing twinning formation as the main deformation mechanism [27]. Furthermore, it is generally accepted that in order to make twinning occurs, the SFE of the steel must be in the range of $18 \text{ mJ m}^{-2} < \text{SFE} < 35 \text{ mJ m}^{-2}$ [28]. In addition, it has been proven that cold deformation led to the grain refinement of $32.39 \mu\text{m}$ and $27.25 \mu\text{m}$ for CR10 and CR20 samples, respectively. This grain refinement will promote the grain strengthening mechanism according to the Hall-Petch relationship. This argument was supported by grain size measurement which reveals finer grain size with increasing the deformation degree. However, the increase in strength of AMS was followed by a significant decrease in ductility in CR10 and CR20 sample by 28.78% and 20.34%, respectively.

In comparison to cold rolled samples, there was a reduction in strength after flash annealing process, followed by a substantial increase in ductility up to 45.71%. Both of yield and tensile strength have decreased to 429 MPa and 869 MPa for CR10FA90 sample and gradually decreased to 388 MPa and 857 MPa for CR10FA150 sample. Similarly, this trend can also be observed in CR20FA90 and CR20FA150. According to the previous microstructure analysis, this change in mechanical properties was associated to the phenomenon of microstructure restoration, which manifested as a decrease in twinning deformation intensity during the recovery stage in FA for 90 seconds and recrystallization stage during FA at 150 second holding time. In addition, it is also important to note that the strain degree has a role in the increment of strength during FA process. At the same holding time, the 20% cold-rolled samples (CR20FA90 and CR20FA150) experienced a higher strength than the 10% cold-rolled samples (CR10FA90 and CR10FA150). However, the strength enhancement is not accompanied by a ductility decrement, which improved by 3–4%. As a result, the best combination of cold deformation and annealing process was achieved in CR20FA150 samples which results the most optimum combination of tensile strength of 860 MPa and a ductility of 45.71%. This result is in line with previous study [15] which reported that combination annealing following cold deformation resulted to the recovery and partial recrystallization microstructure and was able to maintain good strength and ductility leading to high toughness. However, the smaller

density of annealing twin during annealing process is obtained. It was ineffective in maintaining tensile properties. According to Li et al. [29], the high density of the annealed twins and grain refinement are the two parameters that can improve the tensile properties.

Figure 6 shows the value of hardness (HBN) for all samples with different treatment processes. The ST process resulted in a decrease in hardness from 222 HBN to 195.5 HBN. As previously described, the carbide dissolves into the austenite matrix during the solution treatment process, thus lowering the hardness. Furthermore, as the degree of deformation increased from 10% to 20%, the hardness value gradually increased as well, exhibiting a notable rise of 320 HBN in CR10 and 452.9 HBN in CR20. Based on the results of the grain measurements in Table 3, the cold deformation process can refine the grain for both of CR10 and CR20, thus it contributes to the grain strengthening mechanism. When the material is subjected to indentation loads during hardness testing, the formation of DT in the microstructure also inhibits the movement of dislocations, leading to high hardness value.

Subsequently, following the FA process, the hardness value sharply reduced. Like the prior analysis of tensile characteristics, the decrease in hardness value was also contributed by lower intensity of DT during the annealing process, which is in agreement with [30]. On the one hand, at the same degree of strain, the longer annealing time led to a decrease in the hardness values from 222.6 HBN to 214.6 HBN for 10% cold rolled samples and 228.9 HBN to 219 HBN for 20% cold rolled samples. On the other hand, the larger degree of strain prior to FA process had an impact on enhancing the hardness value after annealing process at the same holding time.

Based on Figure 7, it is apparent that longer annealing time and higher degree of strain plays an important role in grain refinement. The recrystallization process began during the 150-second annealing time, which resulted in the formation of new grain nucleation and generated to smaller grains [23], [22]. In addition, the previous deformation process with a higher degree of strain will also produce a number of stored energies, which act as a driving force (motivating force) towards a higher recrystallization rate [15], [23] and encourage the formation of finer grains [31]–[32]. However, in this study, the grain refinement resulted after FA process does not give effect to enhancement of both tensile properties and hardness. This is also in agreement with previous study [33] which suggested that the austenite grain refinement might not be very effective to increase the yield stress of the AMS, since FCC metals are known to have lower values of the Hall-Petch coefficient.

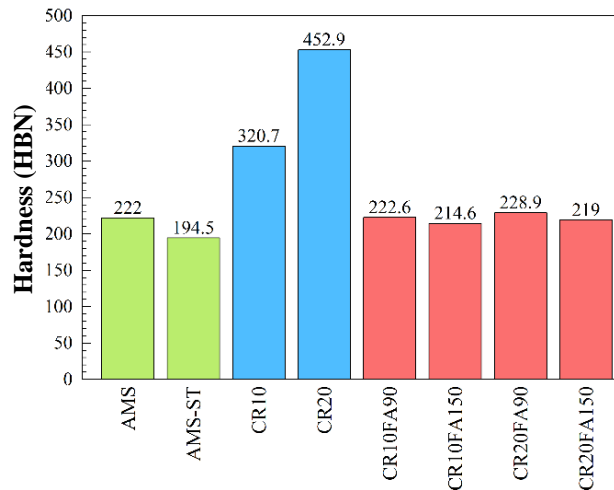


Figure 6. Hardness result of AMS in different treatments processes

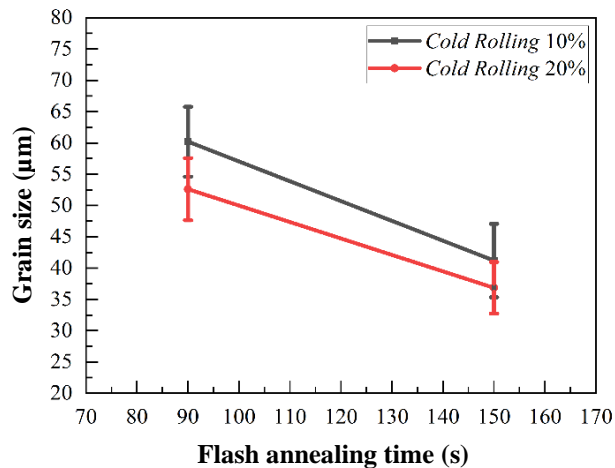


Figure 7. Influence of FA time and degree of strain to grain size

CONCLUSION

According to the study results, applying cold deformation to austenitic manganese steel has considerably increased the mechanical properties, including strength and hardness properties, while was also followed by lowering the ductility. The formation of deformation twin (DT) and annealing twin (AT) during cold rolling have an important role to improve their work hardening capacity, which higher amount of DT were achieved by higher deformation degrees. It will also control the strengthening mechanism. Furthermore, applying flash annealing after cold deformation has successfully improved the ductility due to microstructural restoration. The first stage of restoration starting by recovery stage which manifest with decreasing the intensity of DT and was characterized by formation of partial recrystallisation grains. Furthermore, higher degree of strain will increase the hardness value after FA at the same holding time. On the other hand, the longer annealing time led to a decrease in the hardness value at the same degree of strain. Moreover, both of higher degree of strain and longer annealing time during flash annealing contribute to grain refinement, even though does not affect the increase of tensile and hardness. The most effective combination of cold deformation and flash annealing process was achieved in CR20FA150 sample with a tensile strength of 860 MPa and a ductility of 45.71%. The combination of high strength and high ductility will contribute to the high toughness of AMS which beneficial for high workload applications.

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