Surface martensitization of Carbon steel using Arc Plasma Sintering

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Abstract. In this paper new technology of surface structure modification of steel by short plasma exposure in Arc Plasma Sintering (APS) device is presented. APS is an apparatus working based on plasma generated by DC pulsed current originally used for synthesizing materials via sintering and melting. Plasma exposure in APS was applied into the specimens for 1 and 3 seconds which generate temperature approximately about 1300-1500°C. The SUP9, pearlitic carbon steel samples were used. The hardness, hardening depth and microstructure of the specimens have been investigated by Vickers micro hardness test and Scanning Electron Microscopy (SEM) supported by Energy Dispersive X-Ray Spectroscopy (EDX). The results have showed that the mechanical property was significantly improved due to the formation of single martensitic structures as identified by SEM. The hardness of treated surface evaluated by Vickers hardness test showed significant improvement nearly three time from 190 VHN before to 524 VHN after treatment. Furthermore, EDX confirmed that the formation of martensite layer occurred without altering its composition. The APS also produced uniform hardened layer up to 250 µm. The experiment has demonstrated that arc plasma process was successfully improved the mechanical properties of steel in relatively very short time.

1. Introduction
Various methods to attain the desired surface hardness of carbon steel generally differ in the way the surface is brought to elevated temperature. Surface hardening is generally used to improve tribological properties such as wear resistance and surface fatigue but maintain its ductility [1]. Different processes have own pros and cons. Using ordinary methods for instance flame hardening [2], [3], rapid cooling in quenching medium is required. Temperature of flame hardening process is difficult to control [2], since hardening’s result is controlled by the distance and exposure time. While duration time for induction hardening process is relatively long [4], [5]. The current methods such as electron beam [6] and laser modification hardening [7], [8] enabled a very rapid heating of the surface area above the austenitic temperature.

Selective surface hardening methods such as induction hardening, electron beam, arc and plasma process were used to enhance surface layer without affecting the bulk of the workpiece. Thermal energy absorbed by surface layer was quickly distributed to the entire workpiece, generate very large temperature gradients. This phenomenon lead to self-quenching mechanism resulted in a fine-grained martensitic structure and/or retained austenite [9] with very high hardness. The hardness and the depth of hardened layer vary in in different process, for example in the induction process of
steel the hardness reported as high as 650-700 HVN [4], while in plasma hardening and electron beam the hardness improve significantly, 130-150 % [6, 7].

In this study, Arc Plasma Sintering was used to improve the mechanical properties of SUP9 metal. The APS generally used to synthesize powder into bulk material. The APS process operates at very short time and expected to produce little distortion, induced less residual compressive stresses on the surface. The objectives of this work were to improve mechanical properties of the samples and study its microstructure before and after arc plasma hardening process.

2. Experimental Detail

The samples of JIS SUP9 (leaf spring steel), Table 1 [10], were cut into rectangular shape, 15x15x5 mm (w x l x t). Sample was then grinded as shown in Figure 1 and ready for hardening process. The APS is powered by 12 Volt and 80 Ampere DC current to form electrical arc within Argon gas. The electrically ionized Argon gas (plasma) will exit through the nozzle and applied onto the surface of the samples statically, the nozzle nor samples were not moved. Figure 2 depicts the APS mechanism where temperature of process reached approximately 1300-1500 °C. The Argon gas flow with the velocity of 10 L/minute.

| Material | C   | Si  | Mn  | P   | S   | Cr  | Cu   |
|----------|-----|-----|-----|-----|-----|-----|------|
| SUP 9    | 0.53| 0.31| 0.71| 0.023| 0.012| 0.67| 0.14 |

Figure 1. Grinded sample of SUP9

The arc plasma was applied on the surface of SUP9 samples in three different exposures’ time 0.5, 1 and 3 second, respectively. After surface hardening process, samples were cut in half, cleaned and polished. Two halves of samples were adhered side by side (Figure 3) and mounted into cylindrical resin and fine polished, followed by etching in 2 % nital. The samples were then observed under Scanning Electron Microscope (SEM) to expose the microstructure. Energy Dispersive X-Ray Spectroscopy (EDX) were used to determine the composition of samples, to resolve whether the composition will alter or not. The hardness of the treated layer was evaluated using Vickers micro hardness. The points of hardness measurement were approximately selected as shown in Figure 4.
3. Result and Discussion

3.1. Microstructure of samples

Figure 5(a) and 5(b) below shows the interface between base metal and hardened metal for 1s and 3s exposure, respectively. The figures were obtained from secondary electron imaging (SEI) from SEM. The vivid color depicts the pearlite and ferrite structure and the darker color 5(c) and 5(d) show typical martensite structure. The martensite structure was produced by rapid cooling from austenite phase of samples due to heating by arc plasma.

Figure 3. Two samples were adhered side by side

Figure 4. Hardness measurement (x100)
3.2. Composition and Depth of Hardened Layer

Two samples were selected to examine its composition, 1s and 3 s exposure time respectively. The composition of hardened layer resulted from EDX are depicted in Figure 6. The EDX scanned along the straight path about 70.37µm in first sample and 70.16µm in second sample. Relation of composition of elements in the mass percentage and distance are shown in vertical and horizontal axis, respectively. Fe element has the highest composition in both figures followed by Cr, Si, and C. Figure 6 shows that the composition of its elements was not change along the scanning path. By combining the above result, it concludes that arc plasma process will not alter the composition of the elements but changed its phase and microstructure. The experiment showed that the surface hardness was increased solely due to phase transformation.

![Figure 6. Composition of elements in hardened layer](image)

![Figure 7. Depth of hardened layer (upper 3s, lower 1s exposure, respectively)](image)
The depth of hardened layer in Figure 7 (x35 magnification) shows that 3s exposure resulting deeper layer (Figure 7(b)) compare to 1s exposure (Figure 7(a)). There are two layers in both cross section where the label A is the hardened layer while label B is the heat affected zone (HAZ). Depth of hardened layer of 1s exposure obtained from carefully compare with the magnification scale is about 100 µm while the 3s exposure yield the depth of 250 µm. It concludes that exposure time affected the depth of hardened layer, the longer the exposure time the deeper the hardened layer.

3.3. Hardness

The hardness of specimens was measured using Vickers hardness test at four-point locations as shown in Figure 4. Each sample was hardness tested four times for approximately at the same point location and the results were recorded as in Table 2. Point 1 and 2 are the point at hardened metal, while point 3 and 4 are base metal. The average VHN value then be plotted in Fig.8. At point 1 the average hardness is about 433-524 VHN, slightly lower than the hardness at point 2. At point 3 and 4, where the metal was not affected by heat, the hardness is about 190-199 VHN. The result shows that hardness of hardened layer increased nearly 3x higher compare to base metal.

| Sample 1 | VHN   | Avg. VHN | Sample 2 | VHN   | Avg. VHN |
|----------|-------|----------|----------|-------|----------|
|          |       |          |          |       |          |
| 1 second |       |          |          |       |          |
| 447.7199 | 435.5751 | 433.5751 | 518.6831 | 513.5224 | 513.5224 |
| 395.6053 | 457.4001 | 474.2458 | 503.2009 | 526.935 | 526.935  |
| 481.2457 | 476.5791 | 474.2458 | 526.935 | 526.935 | 524.0234 |
| 197.1182 | 198.0491 | 185.6108 | 215.994 | 199.6476 | 199.6476 |
| 203.7756 | 190.8555 | 185.5908 | 192.7025 | 192.3295 | 192.3295 |

Figure 8. Hardness measurement of samples

4. Conclusion
This experiment provides some significant result in the arc plasma surface hardening process of SUP9 material and potentially can be used for others steel. The results showed that hardness of steel increase nearly three time higher, from 190 VHN in base metal to 524 VHN in hardened layer. Hardness of
surface layer increased due to the formation of martensite phase as observed under SEM. EDX confirmed that the composition of elements (Fe, Cr, Si, and C) remains constant, there was no change in mass percentage before and after hardening process. Depth of hardened layer that have been obtained from Vickers hardness test increased as exposure time increased from 1s to 3s, 100 µm to 250 µm respectively. In summary, arc plasma surface hardening process has demonstrated has significantly improved mechanical properties of steel in relatively very short time.

5. References
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