Investigation of Surface Hardness of Steels in Cyanide Salt Bath Heat Treatment Process

S.P Ayodeji, T.E Abioye and S.O Olanrewaju

Abstract - Carbonitriding of four types of steels, namely, low carbon steel (LCS), medium carbon steel (MCS), low alloy steel (LAS) and high alloy steel (HAS) was achieved using cyanide salt bath heat treatment process. The steels, after preheating, were heated in a diesel fired salt bath heat treatment furnace and then quickly quenched in different media including air, oil and brine. The heat treatment processes were performed at different temperatures ranging from 790^{o}C to 920^{o}C. The heat treatment time was varied from 30minutes to 120 minutes at an interval of 30 minutes so as to investigate its effect on the surface hardness of LCS. The surface hardness of all the heat treated steels samples were measured using an Indentec Rockwell hardness tester. The result revealed that the surface hardness of the heat treated steels is related to the heat treatment temperature and the quenching medium used. Also, the heat treatment time of LCS in salt bath furnace has a significant effect on the surface hardness of the steel.

Keywords: Carbonitriding, Heat treatment temperature, Heat treatment time, Surface hardness.

I. INTRODUCTION

Steels owe their dominance of the field of engineering materials to their ability to provide appropriate properties at economic cost on a production scale. However, the low wear resistance and hardness properties of steels have restricted their use in the engineering field [1]. Existing established methods of improving the surface hardness of steels includes carburizing, nitriding, boronizing and carbonitriding or cyaniding [2], [3], and [4]. These methods change the chemical composition of steels, carburizing by the addition of carbon, nitriding by the addition of nitrogen, boronizing by the addition of borides and cyaniding by the addition of both carbon and nitrogen [2],[3].

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Compared with carburizing and nitriding, Selcuk, Ipek and Karami [3] revealed that carbonitriding treatment is an effective surface hardening method for low carbon and low alloy cold working steels. Though boronizing is the most effective thermochemical treatment for all ferrous materials, the boronized layers are shallow and brittle [3]. Also, the presence of alloy element reduces the diffusivity of boron in the steel and consequently decreases the thickness of the borided layer [3].

Recently, cyanide salt bath heat treatment is preferred over other conventional methods of improving the hardness and wear resistance of steels because of the circulation of molten salt which provides uniform temperature throughout the charge giving accurate temperature control and uniform results, low surface oxidation and decarburisation, and most times parts treated in the salt bath have a clear bright-scale free finish [4]. More so, the inherent cracks and distortions of conventional heat treatment method are eliminated through controlled cooling in molten salt while rapid rate of heat transfer by conduction and convection modes enables a high output to be obtained from a relatively low investment and costs [4].

Specifically, cyanide salt baths offers case hardening and a quick turnaround of the workpiece due to carbon penetration being faster using this process [4]. The hard surface is developed by heating the steel above the austenitizing temperature in a suitable bath of cyanide salt while the carbon and nitrogen diffuse into the steel surface. Thereafter, the heat treated steel is quenched in mineral oil, water or brine.

Extensive works had been done by many researchers on enhancing the surface hardness of different steels grades using diverse processes including carburizing, plasma nitriding, boronizing, cyaniding, etc [1], [2], [3], [4] and [5]. Nevertheless, this work aimed at investigating the surface hardness of carbonitrided low carbon steel (LCS), medium carbon steel (MCS), low alloy steel (LAS) and high alloy steel (HAS) in different quenching media. The effect of heat treatment temperature and time on the surface hardness of the steels was also determined.

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II. EXPERIMENT PROCEDURE

The test materials used in this work included Ø20 x 30mm round bars of LCS (0.15% C), MCS (0.45% C), 42CrMo4 as LAS and X5Cr-Ni18 as HAS. Other materials are industrial Q8 oil, 5% brine solution (5g of NaCl per litre of clean water) and three cyanide salts with the trade names “Ceconstant-50”, “Ceconstant-80” and “Ceconstant-110”.

The compositional analyses of the salts were first determined. Sodium cyanide (NaCN) concentrate in the ceconstant-80 salt was determined by dissolving 1g of crushed powder of the salt sample in 100ml of distilled water. Silver nitrate was later used to titrate the mixture after lead acetate was added to the salt solution as an indicator. The average burette reading of three repeated experiments was recorded as the NaCN concentrate in the ceconstant-80 salt. Potassium Cyanide (KCN) concentrate was determined following the same procedure however ferrous sulphate was added to the salt solution as an indicator. The NaCN and KCN of ceconstant-50 and ceconstant-80 were determined using the same method.

Following the salts compositional analyses, the steel samples were kept in the preheating air furnace after they had undergone wiring. Meanwhile, the furnace pot filled with dry ceconstant-80 salt was slowly heated to 750^{o}C so as to completely melt the salt at about 725^{o}C. The preheated samples of LCS, MCS, LAS, HAS were transferred to the molten salt bath. The samples remained in the salt bath furnace for 30 minutes when the furnace temperature was at 790^{o}C. Subsequently, samples of each type of the steels were quickly removed from the furnace. A group consisted of a sample of each type of the heat treated steels was quenched in air. Another 2 groups of the heat treated steel samples were separately quenched in oil and brine. The quench hardening experiment was repeated for the heat treatment temperatures of 820^{o}C, 850^{o}C and 920^{o}C respectively. Hardness test was conducted on each of the quenched samples.

To study the effects of heat treatment duration on LCS material, 48 LCS samples were prepared and preheated. or each heat treatment temperature, 12 samples were heat treated in the salt bath furnace. At every 30 minutes, 3 LCS samples were removed and each was separately quenched in air, oil and brine. The hardness of all the samples was measured using a multipurpose Indentec Rockwell hardness testing machine.

III. RESULTS

A. Composition Analysis of Ceconstant Salts

The NaCN and KCN concentrates in the analysed salts are presented in Table 2. The result apparenttly revealed that KCN concentrates increases with increasing the ceconstant grade number while the NaCN content decreases with the grade number. The ceconstant-80 has the lowest amount of impurities with equal NaCN and KCN contents.

B. Hardness Tests

The surface hardness of the heat treated steels samples in different quenching media is presented in Fig 2. Low carbon steel samples exhibited the lowest microhardness while the high alloy steel samples had the highest hardness for all temperature values and quenching media. When quenched in air, the LCS, MCS and LAS showed no clear improvement with rise in heat treatment temperature however the microhardness of HAS as shown in Fig 1a shows a significant linear increment from 45.3HRA at 790^{o}C to 67.7 HRA at 920^{o}C. The result obtained for oil and brine quenching media is illustrated in Fig 1b and 1c, the surface hardness of LCS improved slightly while other steels exhibited considerable percentage increase with the HAS having the most significant percentage increase with increasing temperature.

Table 2: Composition analysis of ceconstant salts

S/N	Cyanide Salt	% NaCN	% KCN	% Impurities
1	Ceconstant - 50	73.81	23.25	2.90
2	Ceconstant - 80	48.57	48.64	2.75
3	Ceconstant - 110	22.05	75.14	2.80

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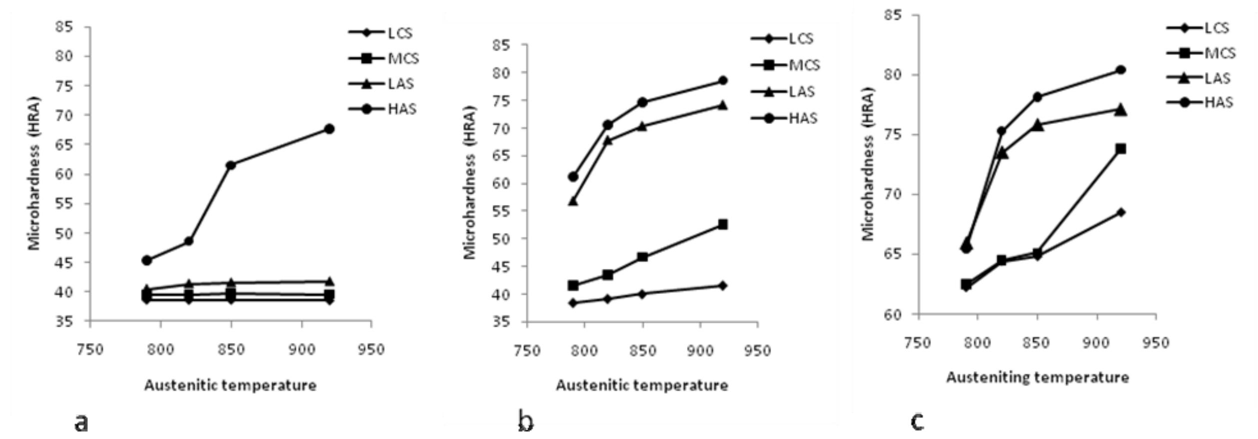

Fig 1: Variation of microhardness with heat treatment temperatures of case-hardened steels quenched in (a) air, (b) oil and (c) brine.

The highest surface hardness of all steels samples at all temperature values was obtained when the heat treated samples were quenched in the brine. The steel samples showed higher hardness values when oil was used as the quenching medium while those quenched in air produced demonstrated the least hardness values.

As shown in Fig 2, the surface hardness of LCS is improved by increasing the heat treatment time. The percentage increase is obvious when the samples were quenched in brine however using air as the quenching medium produced no significant increase. The highest hardness of LCS was achievable when the sample was heat treated (salt bath) at 920^{o}C for 90minutes and then quenched in brine. It is evident in the result presented in Fig 2 that increasing the heat treatment time beyond 90 minutes yielded no noticeable improvement in the hardness of LCS.

The 70.4 HRA surface hardness of LCS at 920^{o}C and 90mins heat treatment time is close to what was obtainable when HAS was heat treated at 820^{o}C for 30minutes and then quenched in brine.

Generally, the surface hardness of cyanide salt bath heat treated steels depends on the choice of quenching medium and the heat treatment temperature. Also, the heat treatment time of LCS in salt bath furnace has a significant effect on the hardness property of the steel.

IV. DISCUSSION

As presented in Table 1, the 50:50 ratio of KCN and NaCN reflects a balanced concentrates of potassium and sodium in the ceconstant-80 salt thus enhancing an equal release of carbon and nitrogen into the surface of the steels [6]. The nitriding (the release of nitrogen into material surface) process is good for increasing the hardness property while carburizing improves the abrasion wear of the materials surface [5]. Consequently, ceconstant-80 enhanced both hardness and wear resistance of the heat treated materials.

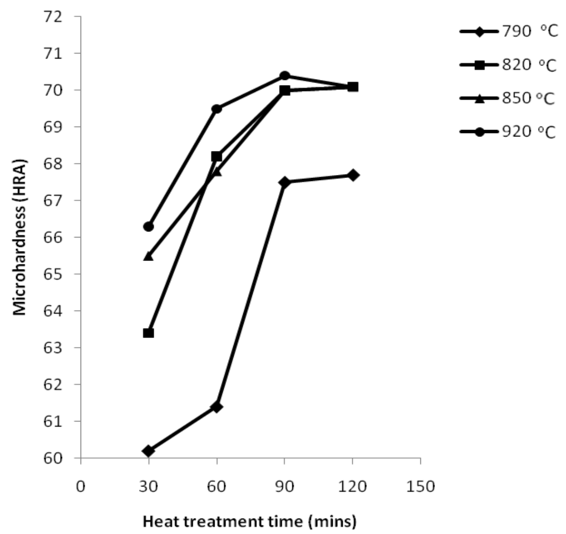

Fig 2: Variation of microhardness with heat treatment duration of low carbon steel

The low surface hardness of LCS samples in Fig 1a, 1b and 1c after quench hardening in different media for all range of temperatures agreed to the fact that low carbon steel does not respond to heat treatment [7]. Likewise, compared to other steels samples, the low carbon content of the LCS samples contributed to the poor hardness result. The better hardness observed in the Fig 1b and 1c is a result of faster cooling experienced by all steel samples in oil and brine media whereas the excellent hardness values demonstrated by HAS samples resulted from the alloying element in the composition of the steel.

At a temperature above 750^{o}C, Na and K, being highly reactive elements, formed different compounds with some elements of steel thereby releasing nitrogen and

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carbon to the skin of the steels. Increasing the temperature may result to releasing of more nitrogen and carbon thereby promoting the formation of more K and Na compounds or oxides. According to Li, Luo, Yeung and Lau [8], the nitrided layer obtained by QPQ salt bath heat treatment can be sub divided into three zones namely; (1) an oxide film zone which is the outer layer (2) a nitrided zone which improves both corrosion and wear resistance properties of the heat treated materials, and (3) the diffusion zone near the base metal. The peak value of nitrogen and carbon concentration is at the middle layer therefore increasing the heat treatment temperature increases the depth of nitrided layer. Consequently, the hardness of the steels increases with increasing the heat treatment temperature.

A clear improvement of the hardness of LCS in Fig 2 is a result of high cooling rate achieved by quenching the steel samples in brine. It is observed that the hardness value at all range of temperature increases as the heat treatment time increases. This is a result of more interaction time which allows for diffusion of more nitrogen and carbon into the middle layer of the hardening case. However, the structure of the nitrided layer became less dense at a time above 90 minutes resulting in negligible rise in hardness of the LCS for the temperature range considered in the experiment.

V. CONCLUSION

This work has successfully investigated the surface hardness of the carbonitrided LCS, MCS, LAS and HAS in air, oil and brine quenching media. The effects of the heat treatment temperature on the surface hardness of the four types of steels and heat treatment duration on the surface hardness of the LCS were fully examined following a logical experimental approach. The following conclusions were drawn from the results obtained.

1. Cyaniding is an effective method of increasing the surface hardness of steels.
2. The surface hardness of cyanided steels depends on the cooling rate experienced in the quenching medium with the brine producing the highest hardness at all temperatures.
3. In all the experiment conducted, HAS followed by LAS demonstrated the highest surface hardness while LCS had the least hardness values.
4. Within the heat treatment temperature range of $790^{o}C$ to $920^{o}C$, the surface hardness of the steels increases with the rise in the temperature.
5. The surface hardness of LCS increases with the heat treatment time however heat treatment time of 90 minutes produced the hardest surface.

REFERENCES

[1] Gui-Jiang Li, Qian Peng, Cong Li, Ying Wang, Jian Gao, Shuyuan Chenc, Jun Wang and Bao-luo Shen (2008), "Effect of DC plasma nitriding temperature on microstructure and dry-sliding wear properties of 316L stainless steel". Journal of Surface Coatings. 202(12), pp2749-2754.

[2] Kumar T., JAmbulingam p., Gopal M. And Rajadurai A. (2004), "Surface hardening of AISI 304, 316, 304l and 316l SS using cyanide free salt bath nitriding process". International Symposium of Research Students on Materials Science and Engineering. December 2002-04 Chennai India.

[3] Selcuk B., Ipek R. and Karami M. B. (2003), "A study on friction and wear behaviour of carburized, carbonitrided and borided AISI 1020 and 5115 steels". Journal of Materials processing Technology. Vol 141, pp 189-196.

[4] Wahl G (1991), "Effect of carbonitriding schedules and subsequent oxidation on the properties of articles". VII International Congress on Heat Treatment of Materials. Vol 33, No 7, pp 487-490.

[5] Suchanek J. and Kuklik V. (2009), "Influence of heat and thermochemical on abrasion resistance of structural and tool steels". Wear, vol 267, pp 2100-2108

[6] Degussa, A.G. (1981), "Handbook on ceconstant 50, 80 & 110: Carrying out the ceconstant process". Hanau 1. Federal Republic of Germany, pp 7 – 9.

[7] Kanwal Tehreem, Nazir Rabia, Zulqernain Saba, Salam A and Ahmad Javaid, "Quench hardening and temperature behaviour of a low carbon steel". Journal of Pakistan Institute of Chemical Engineers, vol XXXVII, pp 51-53.

[8] Li H.Y., Luo D.F., Yeung C.F and Lau K.H (1997), "Microstructural studies of qpq complex salt bath heat-treated steels". Journal of Materials Processing Technology, vol 69, pp 45-49.

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