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Investigation of Ancient Indian Case Hardening Methods by Using Herbals and Plantain Ashes

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Abstract

Iron pillars of Dhar and Delhi, wootz steel swords and many other iron and steel artifacts of our country are testimony to the high level of skill attained by the iron smelters and black smiths of ancient India. In the present paper, it has been endeavored to analyze the status of the ancient metallurgical skill in light of modern knowledge about ancient case hardening methods of Indian blacksmiths. The main objective of present work is to investigate the effect of hardening by using herbals as quenchants and (as carburizing media) pastes. The case hardening pastes were prepared by using sesame oil or ghee, pigeon's & rat's waste, shrungibhasma, plant Arka (jilledu or calotropicgiganta) sap and Tangyedu tree (cassia or seenaauriculta) branches. Banana trunk ash and curd mixer, Banana tree sap, liquid obtained by crushing Banana tree trunk, Banana trunk itself and Ghee are used as quenchents. After completion of heat treatment, hardness, microstructures and chemical compositions are compared with as received sample. Medium carbon steel of grade IS2707 samples are used in this present work. The heat treatment process, hardness, microstructure and spectroscopy investigations were carried out on specimens after heat treatment process. Experimental result shows that microstructure, case hardness values and chemical composition of case of medium carbon steel has changed by given herbal case hardening treatments. The carbon composition introduced onto the surface of the sample results in variation of the hardness and microstructure of the sample.

1. INTRODUCTION

The review of archeological document has produced ample evidence about ancient Indian carburizing methods. Ancient Indians have great historical background in metallurgy. The discovery of iron and the beginning of “iron age” are still controversial but the archaeological evidences as well as the survey of ancient literature have provided it to being at least in the 2^{nd} BC. They also mentioned the possibility of the use of Indian iron chisels and even Indian craft men in the building of the ‘Pyramids’ of Egypt.

The Indian carburizing process has been mentioned in Vedic period. Indians use many techniques such as herbal heat treatment to improve the properties of metals. Indians use herbals and herbal oils in extraction and heat treatment processing of metals & alloys. In many old scripts like Ardhasastra and Bruhusamshitha, Rasasastra mentioned so many processes to harden, strengthen the metal alloys during heat treatment. Especially in hardening they prepare the solutions for quenching to make a metal as hard as to break rocks and improve corrosion resistances.

Different carburizing processes

Naturally, the process must be controlled and the results verified according to specifications, which usually involves case-depth measurement, surface hardness, and microstructural characterization. Depending on the application, retained austenite may be either desired to some amount or may be considered undesirable. Grain boundary carbide networks are universally undesirable. Surface decarburization is also undesirable.

The processes adopted by them can be classified into three categories:

1) Increasing the carbon content of the hot sponge during smelting by raising operating temperature and also increasing the time of retention of sponge iron for slightly longer period, as discussed earlier.
2) Increasing the temperature and time of contact between iron and hot charcoal during secondary refining.

3) Application of a carburization paste on the edge of the sword, etc. to obtain a hard cutting edge with flexible body. Since process (1) and (2) increased the carbon content of the iron rod itself or of the whole mass, this selective carburization process was developed.

Susruta has mentioned the process of carburization by applying a carbonaceous past on the edge of his surgical knives and heating them to red hot condition followed by hardening and tempering treatment to develop razor sharp edges (700 B.C). Varahmihira (~500 A.D) has mentioned the following processes for carburizing and hardening of iron swords:

i) Making a paste with the juice of the plant Arka (calotropis gigantean), the gelatin from the sheep's horn and pigeon' and mouse dung , applying this paste to the steel after rubbing it with sesame oil, heating the sword in the fire and when it is red hot sprinkling on it water or milk of mares (camel or goat) or ghee(clarified butter) or blood or fat or bile and then sharpening on the lathe.

ii) Plunging the steel red hot into a solution of plantain ashes in whey, keeping it standing for twenty four hours, and the sharpening on the lathe. (varahamihirakaharglakshanam, chap.XVIX, slokas 23-26)

Carburizing is one of the most widely used surface hardening techniques for steel, mainly because it can offer the superior combinations of mechanical properties. The function of the carburizing medium is to release atoms of carbon at the surface of the work piece so that, at the carburizing temperature, they will be absorbed interstitially into the steel [1-3].

2. MATERIALS AND EQUIPMENT

Specimen Details

Case hardening or carburizing applicable for low carbon and medium carbon steels. A chemical compositions have been proposed for the candidate steel in this experiment was medium carbon steel IS2707 Grade 2. Table 1 shows the chemical composition of the steel. Proposed compositions contain lower level of C.

Table 1: Chemical compositions in weight percent for the initial received steels

%	C%	Si%	Mn%	P%	S%
Required	0.55-0.60	1.5-2.0	0.70-1.0	0.05 max	0.05 max

One inch length with 23mm diameter samples are prepared by using lathe machine and these are austenised at 860 ^{o}C and furnace cooled. These samples are used for further experiment.

Materials Required

In this experiment we used several herbals, oils and bio materials for case hardening. All these materials are selected based on the ancient scripts and shlokas in Bruhusamhitha and SusruthaSamhita. Those materials are listed below

- Banana tree trunk
- Banana tree sap
- Tangyedu tree branches
- Curd
- Sesame oil
- Ghee
- Shrungibhasma
- Rats and pigeons dug

Preparation Of Herbal Quenchants And Pastes

Herbal Quenchants

1. Liquid extracted by crushing the banana trunk and left aside for 10 days.
2. Sap extracted from banana trees by cutting the banana fruit plantain.
3. 2 feet length Banana trunk with a hole in center to insert the sample
4. Finely chopped Tangyedu tree branches for packing purpose
5. Normal water
6. Cow Ghee
7. Mixer of curd and banana trunk ashes. (burning the trunk in closed vessel

Herbal Pastes

Type I : This paste was prepared by mixing the Sesame oil with equal quantities of

- Shrungibhasma
- Rats and pigeons dug
- Arka milk

This paste is applied for carburizing the samples. The sample is coated with sesame oil and kept in a small steel box which is half packed with the above paste. Now completely pack the sample with the remaining paste and cover the box with a lid having small holes on it to allow gases to escape. Then these boxes are subjected to heat treatment.

Type II: This paste was prepared by mixing the cow ghee with equal quantities of

- Shrungibhasma
- Rats and pigeons dug
- Arka milk

These pastes applied for carburizing the samples. For this we chose a small steel box and place the sample in it. Then applied the ghee on sample and rubbed. Then the box was paste was completely covered with herbal paste and packed with lid which have small holes on it to allow escaping of gases. Then these boxes are subjected to heat treatment.

Type III: This sample was made with finely chopped Tangyedu tree branches.

Equipments Used

Following equipments are used in this project

1. Furnace
2. Rockwell hardness testing machine
3. Optical microscope
4. Spectrometer

3. EXPERIMENTAL PROCEDURE

Sample Preparation

For the purpose of experiment the hot-rolled steel rods were further machined into 1” length and 23mm diameter specimens in the Machine Shop by lathe machine. Totally 10 samples are prepared with the dimensions as mentioned above. Then all these samples are austenite at 860 ^{0}C with soaking time 1 hour then furnace cooled. Then samples are roughly polished on both sides and surfaces are cleaned to remove the dust and unwanted materials.

Then three samples are separately placed in a steel boxes and then fully filled with

1. The first type paste was prepared by mixing the Sesame oil with equal quantities of Shrungibhasma, Rats and pigeons dung, Arka milk.
2. The second type paste was prepared by mixing the ghee with equal quantities of Shrungibhasma, Rats and pigeons dung, Arka milk.
3. The third sample was placed in a box and filled with Finely chopped Tangyedu tree branches

Heating & Soaking

a) All the specimens (samples with in boxes) were heated to a temperature of 860^{0}c

b) At 860^{0}c the specimen was held for 1 hour

c) Then the furnace was switched off.

The objective of keeping the specimen at 850^{0}c for 1 hr is to homogenize the specimen. The temperature 860^{0}c lies above Ac1Temperature. So that the specimen at that temperature gets sufficient time to get properly homogenized .The specimen was taken out of the furnace was switched off.

Quenching:

1. First sample was quenched in Water or liquid extracted by crushing the banana trunk which was leaved it for 10 days without stirring
2. Second sample was quenched in Sap extracted from banana trees by cutting the banana fruit plantain and soaked for 3days
3. Third sample was placed in a hole created in 2 feet length Banana trunk and leave it for 3days
4. Forth sample, finely chopped Tangyedu tree branches packed sample was taken out and quenched in water.
5. Fifth sample which placed in carburizing paste with ghee was quenched in liquid extracted by crushing the banana trunk.
6. Sixth sample which placed in carburizing paste with sesumn oil was quenched in liquid extracted by crushing the banana trunk
7. Seventh sample was quenched in Norman water.
8. Eighth sample was quenched in Ghee.
9. Ninth sample was quenched in the mixer of curd and banana trunk ashes obtained by burning the trunk in closed vessel.

Metallographic Study

The samples were polished on series of emery paper ranging from 180, 220, 400, 600, 1/0, 2/0, 3/0, and 4/0. Polishing was done at four perpendicular directions. After polishing by emery papers the sample was polished by disc polishing. As abrasive and carrier, alumina powder was used and water to avoid unwanted surface scratches. After disc polishing, the sample was cleaned with acetone. Later the samples were etched with 2% Nital solution reagent to reveal the microstructure. Then the sample was examined under the advanced optical microscope at different magnifications.

Chemical composition

The alloy purchased from Tarak alloy steel suppliers, Vijayawada was used in the present study. The composition of the sample were tested by spectroscopy in Better Castings plt.ltd and G.S Alloys pvt ltd, provide by the manufacture is mentioned in table.

Hardness testing

- The hardness of material is defined as resistance to indentation, abrasion, deformation with applied load.
- The indenter and anvil should be clean and well seated.

- The surface to be tested should be clean and dry , smooth, and free from oxide .a rough ground surface is usually adequate for the Rockwell test.
- The surface should be flat and perpendicular to the indenter.
- Tests on cylindrical surfaces will give low readings, the error depending on the curvature, load, indenter and hardness of the material .theoretical and empirical corrections for this effect have been published.
- The thickness of the specimen should be such that a mark or bugle is not produced on the reverse side of the piece.it is recommended that the thickness be at least 10 times the depth of indentation. Tests should be made on only a single thickness of material.
- The spacing between indentations should be three to five times the diameter of the indentation.
- The speed of application of the load should be standardized. This is done by adjusting the dashpot on the Rockwell tester. Variations in hardness can be appreciable in very soft materials unless the rate of load applications is carefully controlled .for such materials the operating handle of the Rockwell tester should be brought back as soon as the major lad has been fully applied

4. RESULTS AND DISCUSSION

Microstructure

Figure 1 (a) shows optical micrograph (1000 X mgnification) of specimens of as-received samples annealed at 860 ^{o}C. It is evident from the figure that white areas are ferrite and darker region are pearlite. From dark field images (not shown here) of the sample clearly evidenced the variation between ferrite and pearlite. In as received sample the coarser pearlite and ferrite are observed in high quantity.

The micrographs of sample 2 quenched in banana trunk juice shown in Figure 1(b). The microstructure of the sample reveals the retained austenite as white region and darker needle like region as martenisite. Dark field images (not shown here) of these sample shows the retained austenite network is glowing fluorescently in the image surrounding by the martensite regions.

Figure 1(c) describes the microstructure of the sample quenched in banana sap. Figure shows the bright region which can be associated with ferrite and darker region are related to pearlite. The distribution of pearlite and ferrite can be easily seen in the dark field images (not shown here).

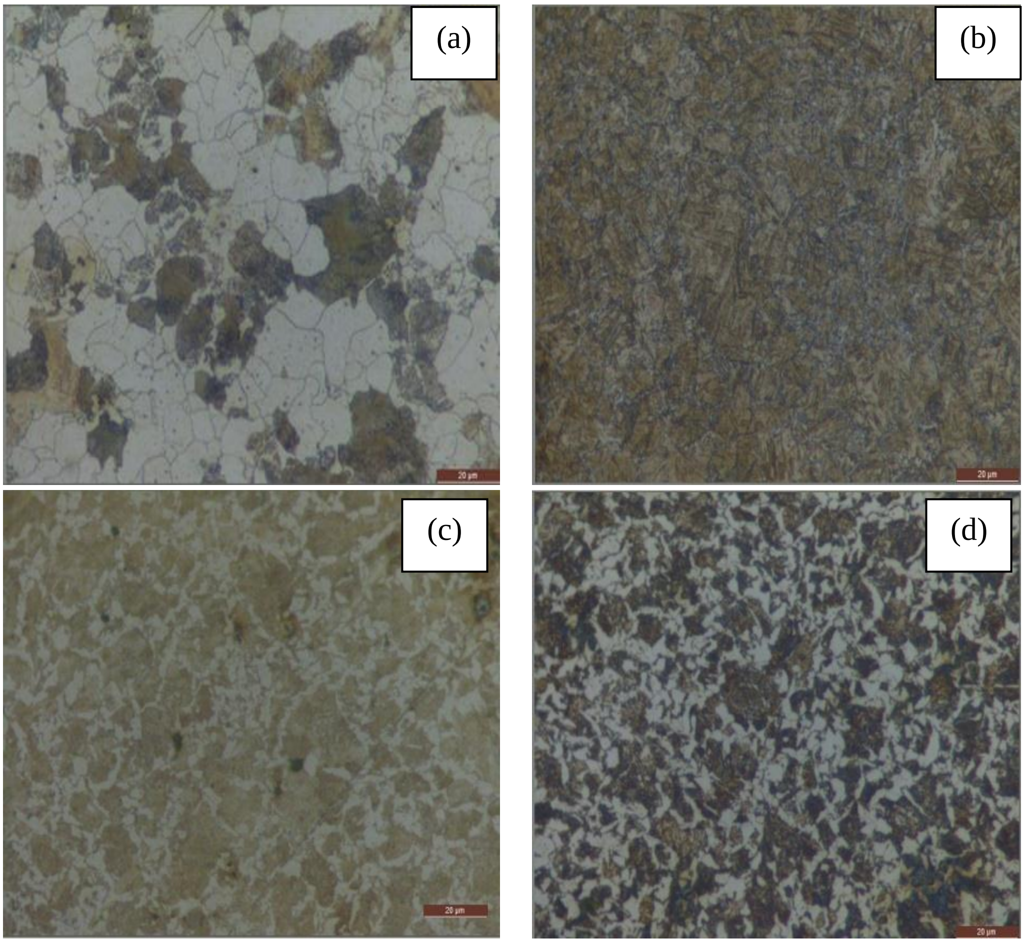
Figure 1 (d) shows the microstructures of sample cooled in banana trunk. Fine grains of pearlite in ferrite matrix is observes in the micrograph. The pearlite grains are observed in the micrograph which are distributed with angular or sharp edged. From dark field images (not shown here) it is clearly visible that the high amount of pearlite network in ferrite.

Figure 1(e) microstructure of sample which treated with tangyedu tree. The micrograph reveals the white regions are related to retained austenite and ferrite. Thick grain boundaries observed in the micrograph may be due to the accumulation of carbon in the boundary regions. From the dark field images (not shown here) it can be clearly evidenced that thick grains is glowing fluorescently in the image.
The microstructure of the sample treated with type-I carburizing paste is shown in Figure 1(f). Figure 1(g) shows the microstructures of the sample coated with type II carburizing paste. From these microstructures, it is evident that very fine grains of small ferrite and retained austenite and darker grain boundaries with high carbon content can be observed from the microstructure.
The micrographs shown in Figure 1(h) is related to the sample quenched in water. The white region observed in the micrographs are related to the ferrite and retained austenite and darker regions are related to the pearlite. It was observed in the dark field images (not shown here) that the retained austenite network is glowing fluorescently in the image.
The microstructure of sample quenched in cow ghee is shown in figure 1(i). White area observed in micrograph are due to retained austenite and darker needle shaped regions are due to martensite, the micrograph also consists of small amount of ferrite.
Figure 1(j) shows the microstructure of the sample cooled in banana trunk ash ans curd mixer. A very fine and sharp grains of pearlite in ferrite matrix was observed in the microstructure. The ferrite grains are with angular or sharp edged.
Table 2 describes the hardness values of the different treated samples. From the table, it can be seen that pack carburized sample with ghee has highest hardness of 67 HR_C on the case while the as received sample hardness is 29 HR_C. This may be due to the microstructural changes observed in the sample which may be generally associated with formation of pearlite.

Table 2. Hardness values of heat treated specimens

S.NO	SAMPLE TYPE	HARDNESS HR_c
1	As Received Sample	29
2	Banana Trunk Juice	64
3	Banana Sap	32
4	Banana Trunk	35
5	Tangyedu Tree Used	60
6	Type I Packed With Ghee	
	Case	67
	Middle	52
	Core	47

7	Type II Packed Packed With Oil	
	Case	58
	Middle	49
	Core	38
8	Water Quenched	58
9	Ghee Quenched	44
10	Curd +Ash Quenched	40



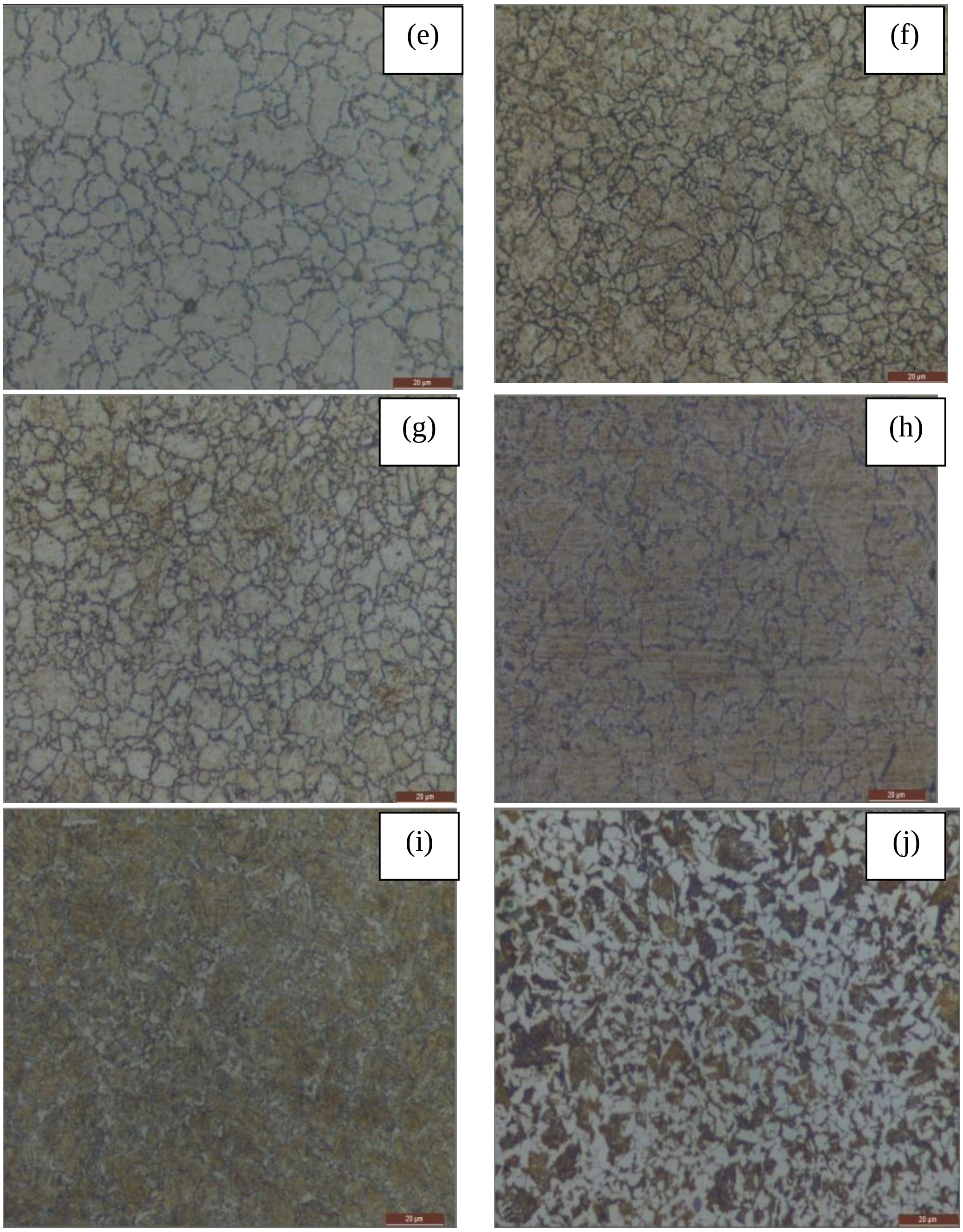


Figure 1. Microstructure of the sample treated with different mediums.

Chemical Compositions

Spectrometric analysis was conducted on all specimens to know the variations in chemical compositions of treated samples. The chemical analysis of samples is presented in table 3.

Table 3. Chemical composition of the treated samples

Sample type	**Major alloying elements**						
	C	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**
As received	0.37	0.28	1.26	0.013	0.04	0.08	0.081
B.t.juice	0.32	0.31	1.31	0.015	0.06	0.08	0.091
B.sap	0.42	0.06	1.93	0.01	0.05	0.09	0.11
Tangyedu	0.41	0.07	2.01	0.017	0.06	0.08	0.09
Type II. ghee	0.533	0.316	1.97	0.017	0.07	0.08	0.08
Type I	0.434	0.306	1.26	0.015	0.05	0.08	0.09
Banana trunk	0.407	0.055	1.97	0.015	0.06	0.09	0.10
Curd+ash	0.388	0.0627	1.99	0.016	0.06	0.08	0.08

The chart gives the variation in carbon percent .The as received sample has 0.37%C while the type II pack carburized sample in ghee has greater carbon content of 0.53%C. The variation of hardness values of the samples treated in different mediums can be associated with the changes in the microstructures which consist of ferrite, pearlite and martensite. The change in hardness values can also be attributed to the diffusion of carbon content present in the sample which was evidenced from the spectrometric analysis [4].

CONCLUSION

The present work focuses about the case hardening of steel using ancient herbals and plantain ashes treatment. The maximum case hardening is obtained by using type I and type II carburizing pastes i.e. 68 and 67 HRC respectively. Samples quenched in banana trunk juice shows peak hardness of 64 HRc because of martensitic formation. Type III carburizing paste showed a hardness value of 60 HRc. Next higher hardness of 58 HRc reported to water quenched sample. From this work, it is clear that ancient case hardening methods are fast and more efficient in all aspects compared to modern carburizing methods. With these quenching mediums, it is possible to produce better products with optimal cost and with superior properties compared to modern methods.

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