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Investigation on Deep Cryogenic Treatment of Brass Powder Compacted Parts

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ABSTRACT
Deep cryogenic treatment has been proved as the most viable and effective method for enhancing the mechanical properties of metals in the recent past. This project aims to investigate the effect of cryogenic treatment on the mechanical properties of brass powder compacted parts. The powder metal part is made by mixing the alloy powders of desired composition, forming the mixture into a desired part shape and sintering it to a desired temperature. The sintered part is then subjected to cryogenic cooling using liquid nitrogen. Experiments are carried out for comparing the variation in the mechanical properties (strength, hardness, and wear resistance) of brass parts after cryogenic treatment with that after conventional heat treatment.

KEYWORDS: *Deep cryogenic treatment, powder compact, sintering, brass powder*

INTRODUCTION

In modern day industry some of the engineering components fail to achieve the desired performance level due to the lack of sufficient quality of materials. In one way or other, properties of materials such as brass are found insufficient to meet the functional requirements of parts used in manufacturing and assembly [1]. Continuous research works are intended to enhance the life cycle of engineering components by seeking alternate process routes. Significant property improvement includes increased hardness, strength and electrical conductivity. If some or all of these property enhancements can be achieved, it should be possible to utilize a more energy efficient manufacturing process to produce better quality brass components at lower costs. Progressive heating of cold worked brass can relieve internal stresses, prevents stress corrosion cracking and minimizes distortion at 250^0C. In the fully annealed condition at 500^0C strengthening effect of cold working is lost and it becomes softer [2]. Cryogenic treatment is an inexpensive add on process over the conventional heat treatment in which the samples are cooled down to the prescribed cryogenic temperature level at a slow rate, maintained at this temperature for a long period and then brought back to room temperature at a controlled rate. The notable expected effects in cryogenic treatment include changes in mechanical properties and microstructure of brass material [6]. Cryogenic treated hard brass wires are used in production of high-performance dies, punches, etc to increase its wear resistance and relieve its residual stresses. After a comparative study it is established that machining Time of Titanium Alloy (Grade2) using Cryogenically Treated Brass Wire is More Effective than normal brass wire [6].

Powder metallurgy process involves mixing, compacting and sintering of the metal powders which permits customization of material properties. This technique allows the creation of net shape components at high production rate [7]. P/M brass parts have special physico-mechanical properties such as high electrical and thermal conductivities, high corrosion resistance and low coefficient of friction. They also possess better toughness, vibration and tribological properties compared to forged parts [10].

Significance of this Research

Cryogenic treatment has proved its worth in enhancing the mechanical and metallurgical properties of metals. Brass is basically a medium strength (280-580N/mm2) engineering material having less wear resistance [2]. Improvement in hardness and wear resistance can contribute significantly to the usage of brass in modern manufacturing industries. The relevance of powder metallurgy lies in its ability to produce near net shape components economically. It produces components with good surface finish and dimensional accuracy. Greater control of material properties can also be achieved.

Literature Survey

This chapter presents the literature review on the research works conducted in this field. Specific focus is given on identifying the influence of conventional heat treatment, deep cryogenic treatment and powder metallurgical processes on Brass.

Conventional heat treatment (Annealing)

Annealing is a process wherein heat is applied to a metal in order to change its internal structure in such a way that the metal will become softer.

Khan et al. [1] says annealing is done to achieve the specified characteristics such as hardness, and grain refinement. It induces ductility as well as softness and also relieves the residual stresses that are developed during various machining processes. Brass becomes stronger and more corrosion resistant with the addition of small percentage of Aluminum. Vin Callcut [2] has reported that on progressive heating, the internal stresses in cold worked brass will be relieved at 250^{o}C. Thus, annealing prevents stress corrosion cracking and minimizes the amount of distortion which may occur during machining. At 400^{o}C some fundamental change occurs and above that the material starts to anneal or soften with time. Brass softens when heated to 500-550^{o}C for 1⁄2 to 1 hour at that temperature and then air cooled.

Deep Cryogenic Treatment (DCT)

DCT is the ultra-low temperature processing of materials to enhance their desired metallurgical and structural properties. The Cryogenic treatment affects the micro structure of the material which in turn enhances the properties of the materials such as hardness and wear resistance.

Baron [4] has reported that keeping the brass wire in cryogenic environment, improves its wear resistance and relieves residual stresses. According to Kim et al. [5] cryogenic treatment of Ni-Ti alloy recorded an in increase in micro-hardness. There was no measurable change in elemental or crystalline phase composition. Kapoor et al. [6] have carried out experimental work on brass wire electrodes using deep and shallow cryogenic treatment and observed that grains are more refined than that in non-treated electrodes. Also, a marked improvement in the electrical conductivity of these electrodes is noted after deep and shallow cryogenic treatment.

Powder metallurgy

Powder metallurgy is a manufacturing process where various powder metals such as stainless steel, brass, copper, iron and bronze are compressed and then sintered (heated) for bonding the particles metallurgically. This remarkably "green" and environment friendly process uses 99% of the material, that's in the mold, producing harmless bi-products of nitrogen and hydrogen.

Radomysel'skii et al. [7] says the best processing properties are exhibited by brass powders manufactured by the diffusional impregnation technique, using a zinc powder, brass swarf or a copper-zinc master alloy as a point source. As per Raymond [9] the invention throws light into the production of improved brass objects of accurate configuration having physical properties superior to those heretofore obtainable in brass objects formed by compression and heat treatment of metal powders. R. Z. Vlasyuk et al. [11] detected the formation of a defective structure zone in its surface layer during heat treatment. Absence of the beta-phase and higher porosity are found in this zone due to the evaporation of zinc from brass during sintering. To prevent this zinc loss, brass powder compacts are heated in the temperature range 873-I173^{0}K

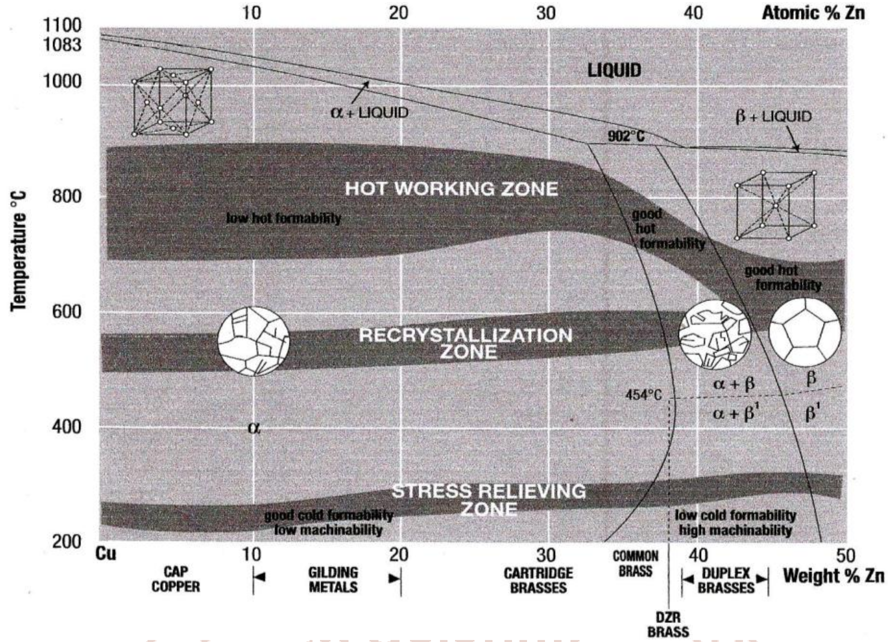

Fig.1 Copper-Zinc partial phase diagram (Vin Callcut1996)

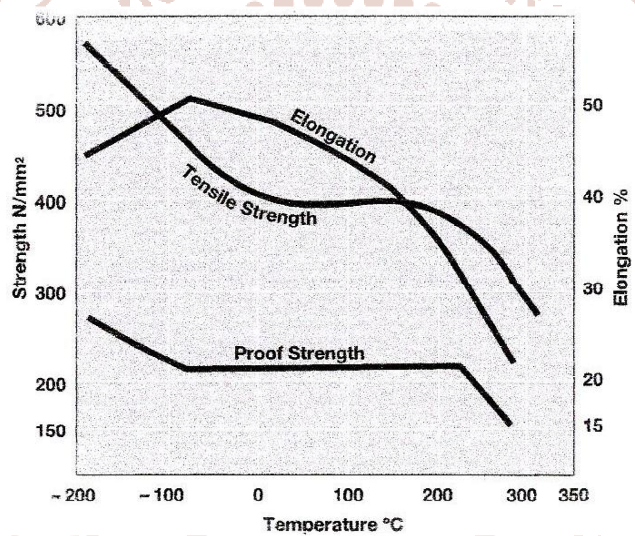

Fig-2 Effect of temperature on free machining brass (VinCallcut1996)

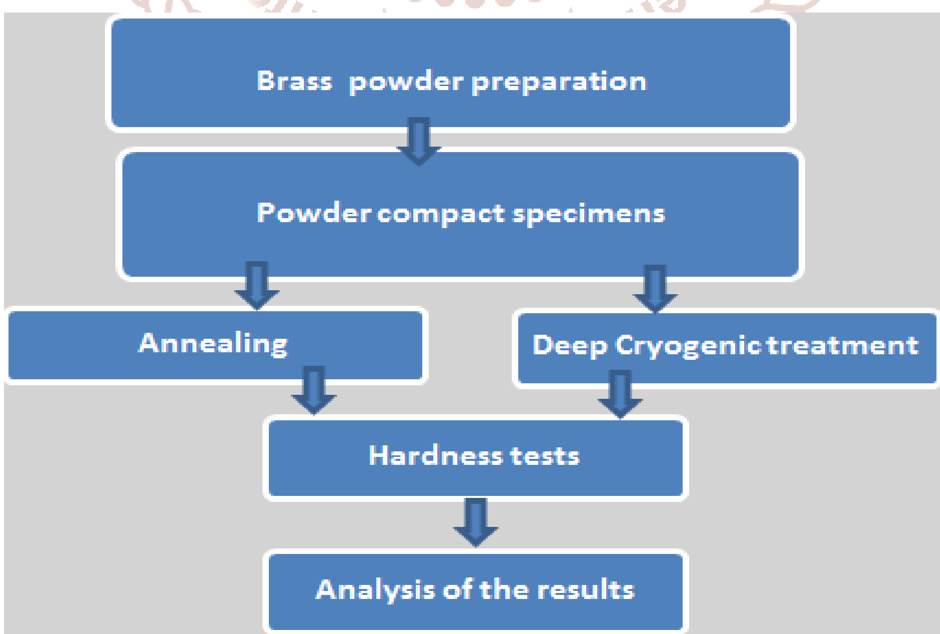

Fig.3 Experimental Program

Powder compaction

The standard specimens for experimental work are made as per ASTMB925-03, B331, B823 and E92-17 by powder compaction and sintering. Prepare the powder mixtures of three different compositions of Cu and Zn (% by weight-alpha alloy) and compacted them in a power press at a pressure of 200MPa. The powder, preparatory to compression in the mould, has to be mixed in a non-oxidizing atmosphere for two hours. Thorough mixing is required and a rotary mixer turning at high rpm produces the necessary degree of homogeneity of the powder in the two-hour period. The compacts will then be sintered for one hour in muffle furnace to a temperature of 450^0C at 7.5^oC per minute, held at that temperature for another 15 minutes and thereafter cooled in such atmosphere prior to exposure to the air. The powder compacted parts obtained then can be cut into desired length and taken for further heat treatment processes. Three types of specimens are prepared with different combinations of Cu, Zn and Al as shown in Table1.

Table1. Specimen details

Specimen No.	Powder Composition wt. (%)	Compaction Pressure (MPa)	Sintering		Number of Samples	
			Temp. (^{0}C)	Soaking Time (Minutes)	Furnace Cooled	Air Cooled
1	Cu-70, Zn-30	200	450	15	3	1
2	Cu-65, Zn-35	200	450	15	3	1
3	Cu-67, Zn-32, Al-1	200	450	15	3	1

Specimen sizes

A. D=20mm., L=25mm.
B. D=15mm., L=20mm.

Annealing

One specimen from each lot is subjected to annealing after sintering. Heat the powder compacted brass specimen to 600^oC at 10^oC per minute, keep it for 1⁄2 hour at that temperature and then furnace cooling is done [3]. To ensure that excessive grain growth is prevented by a quench or rapid furnace cool. 'Flash' annealing may be carried out at higher temperatures for considerably shorter times, but measures have to be taken to avoid excessive grain growth. The use of a protective atmosphere reduces oxidation. Normally this can be prepared from cracked or partly burnt ammonia to give an atmosphere high in nitrogen and water vapor. Since zinc is volatile, care needs to be taken to avoid overheating. Parameters such as time and temperature can be varied to check the changes in properties. As brass usually melts at around 900^0C, necessary steps can be taken to avoid overheating. The process details are shown in Table2.

Table2. Annealing

Specimen No.	Number of Samples	Powder Composition wt. (%)	Annealing		Furnace Cooling Rate/minute	Soaking Time (minutes)
			Temp. (^{0}C)	Heating Rate/minute		
1	1	Cu-70, Zn-30	600	@10^0C	@4^0C	30
2	1	Cu-65, Zn-35	600	@10^0C	@4^0C	30
3	1	Cu-67, Zn-32, Al-1	600	@10^0C	@4^0C	30

Deep cryogenic treatment (DCT)

After the compaction, brass specimens of standard size were subjected to deep cryogenic treatment using liquid N_2 in a vacuum chamber [6].

The experiment was conducted in three stages,

1. Ramped down to -190^0C in nine hours (cooling rate approx. 0.4^0C/min.)
2. Soaked in liquid N_2 at that temperature for eighteen hours
3. Ramped up for nine hours (heating rate approx. 0.4^0C/min.)

The slow cooling process from ambient to cryogenics is important in avoiding thermal stress. Cryogenic treatment changes the entire structure of metal, not just surface. Table3 shows the experimental details.

Table3. Deep Cryogenic Treatment

Specimen No	Powder Composition wt. (%)	Deep Cryogenic Treatment			Number of Samples	
		Ramp rate approx. (^{0}C/minute)	Temperature (^{0}C)	Soaking Time (Hours)	Furnace cooled	Air cooled
1	Cu-70, Zn-30	0.4	-190	18	1	1
2	Cu-65, Zn-35	0.4	-190	18	1	1
3	Cu-67, Zn-32, Al-1	0.4	-190	18	1	1

Hardness Test

Hardness is defined as the resistance of a material to various kinds of permanent shape change and penetration by another harder material.

Vickers hardness testing machine is used to conduct the test. It is an indentation test in which a Vickers square-based pyramidal diamond indenter having specified face angles is forced under specified conditions into the surface of the test material, and, after removal of the test force, the lengths of the two diagonals of the projected area of the indentation are measured to calculate the Vickers hardness number. *Vickers hardness number*, HV, *n*—the calculated result from a Vickers hardness test, which is proportional to the test force applied to the Vickers indenter divided by the surface area of the permanent indentation made by the indenter after removal of the test force. The surface area of the permanent indentation made by the Vickers indenter is calculated partly on the measured mean length of the two diagonals of the projected area of the indentation [8]. Details of the hardness tests are shown in Table5. The repeatability is estimated as the percent range of *n* diagonal measurements with respect to the measured average hardness value as:

$R = 100 \times [(d_{max} - d_{min}) / \bar{d}]$

Where:

R = Repeatability,
d_{max} = the longest diagonal length measurement made on the standardized test block,
d_{min} = the shortest diagonal length measurement made on the standardized test block, *and*
$\bar{d}$ = the average of the *n* diagonal length measurements made on the standardized test block

Calculation of the Vickers Hardness Number
$HV = 0.1891F/(d)^2\ N.mm^{-2}$

Where:
d = *average length of diagonals, and*
F = *Test force applied*

Equipment Used: Micro Hardness Testing Machine.
Load: -200g.
Dwell Time: -10 Seconds

Results and Discussion

This analysis and discussion of the results obtained from the experimental work conducted in powder metallurgy and deep cryogenic treatment. The influence of cryogenic treatment and effect of annealing on the material characteristics and mechanical properties of powder compacted brass specimens are done. Variation in properties of furnace cooled and air-cooled specimens having different compositions have been discussed in this chapter.

Effect of Deep Cryogenic Treatment on Powder Compacted Brass Specimens

The experiments are conducted to find the effect of deep cryogenic treatment on the mechanical properties of powder compacted brass specimens and the results are used to analyse and compare with that of the annealed specimens.

Hardness

The experimental data for Vickers Hardness of the untreated, deep cryogenically treated and annealed powder compacted specimens are presented in Table 5. Based on this data, the bar chart for hardness is plotted. Variation of hardness in specimens having different compositions before and after heat treatment processes is shown in Fig.10. It can be seen from the figure that hardness values obtained with untreated and annealed specimens are lower than that of deep cryogenic treated powder compacted specimens. It is also observed from the fig. that hardness of furnace cooled specimens possesses highest hardness values on cryogenic treatment. Annealed specimens are found to have lowest hardness among the three. Hardness is also found to decrease with the decrease in percentage of Cu. Specimens with small percentage of aluminum in them recorded hardness values lower than that of pure brass specimens.

Table 4 Hardness Test Results

Sample Name	Cryogenic Treated		Untreated		Annealed	
		Mean HV		Mean HV		Mean HV
Sample1 Cu-70, Zn-30	69.2 74.7 69.4 70.8 72.6	71.3	62.5 61.9 59.3 63.4 58.4	61.1	59.7 63.8 68.7 60.1 69.3	64.3
Sample2 Cu-65, Zn-35	79.2 77.2 66.3 79.3 65.2	73.4	60.4 61.5 59.5 62.8 69.2	62.7	62.3 69.2 65.3 76.8 69.5	68.6
Sample3 Cu-67, Zn-32, Al-1	75.2 68.9 70.6 73.2 69.7	71.5	59.2 63.8 59.1 64.9 65	62.4	59.6 61.2 59.1 68.8 73.7	64.5

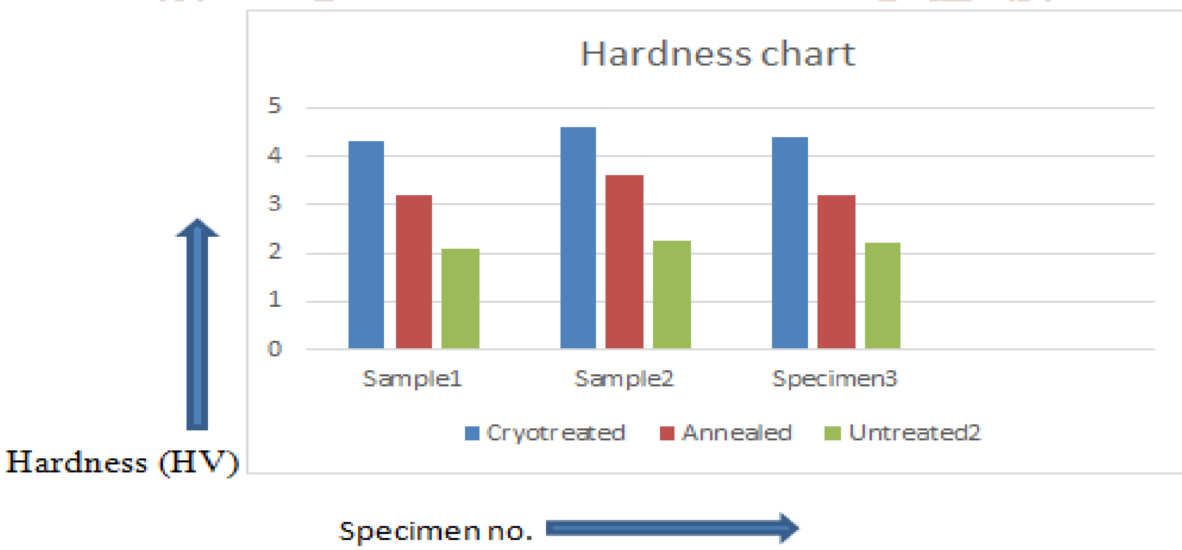

Fig.4 Comparison of hardness between treated and untreated specimens

It is observed from Fig.4 that cryogenic treated specimens have higher hardness values than annealed and untreated specimens.

Table 4 Hardness comparison after DCT and Annealing

Sample Name	Vickers Hardness number HV			Variation in Hardness after DCT (%)	Variation in Hardness after Annealing (%)
	Untreated	Cryogenic Treated	Annealed		
Sample1 Cu-70, Zn-30	61.1	71.3	64.3	16.69	05.24
Sample2 Cu-65, Zn-35	62.7	73.4	68.6	17.07	09.41
Sample3 Cu-67, Zn-32, Al-1	62.4	71.5	64.5	14.58	03.37

SEM analysis

SEM was carried out for DCT, annealed and untreated samples to study the micro structural changes. The results showed nanoprecipitation in cryogenically treated specimens. Dark region in annealed and untreated samples shows Zn depletion by oxidation. Results of the SEM analysis are shown in Fig.5, Fig.6 and Fig.7 for DCT, annealed and untreated brass samples respectively.

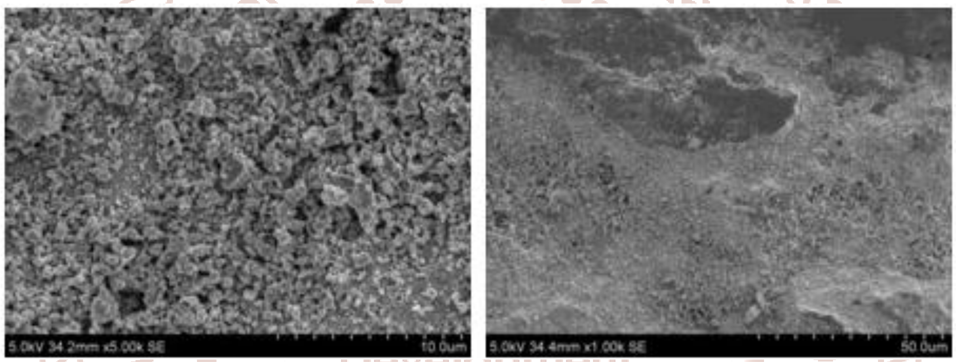

Fig.5 Results of SEM for cryogenically treated samples

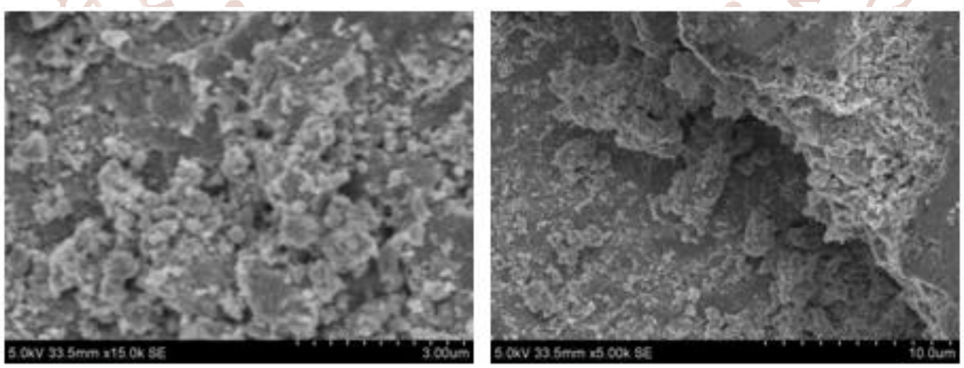

Fig.6 Results of SEM for annealed samples

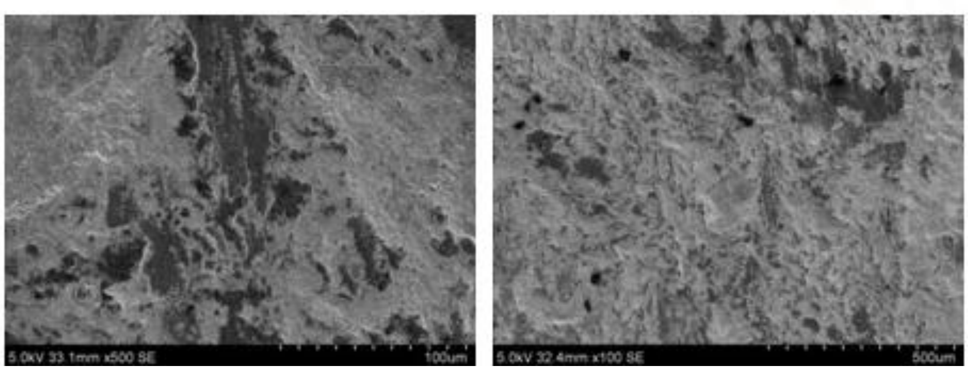

Fig.7 Results of SEM for untreated samples

Conclusions

The investigations into Deep Cryogenic Treatment process on powder compacted brass parts for exploring the possibility of improvement in its mechanical properties, achievement of material efficiency and cost reductions have led to useful results. The important conclusions have been listed below

1. Hardness is found to be increased by 16.69%, 17.07% and 03.37% for specimen1 (Cu-70% & Zn-30%), specimen2 (Cu-65% & Zn-35%) and specimen3 (Cu-67%, Zn-32% & Al-1%) respectively after cryogenic treatment.
2. Hardness is found to be increased by 05.24%, 09.41% and 14.58% for specimen1 (Cu-70% & Zn-30%), specimen2 (Cu-65% & Zn-35%) and specimen3 (Cu-67%, Zn-32% & Al-1%) respectively after Annealing.
3. From SEM analysis, it is evident that nano-precipitation of atoms took place in cryogenic treated specimens. In untreated and annealed specimens, Zn corrosion is visible.

Concluding Remarks

From the research work, it has been concluded that the deep cryogenic treatment has enhanced the hardness of powder compacted brass specimens significantly.

References

[1] N. Z. Khan, A. Khan, A. Shakoor, K. Azam, (2015) "*Manufacturing Defects of Brass Products and suggested Remedies*". International Journal of Innovative Science, Engineering & Technology, Vol. 2, Issue 9.

[2] "*The Brasses properties and applications"* CDA Publication No. 117, (1996) by Vin Callcut Revised 2005 by Peter Webster.

[3] Arun Kumar Verma, PrantikMukhopadhyay, S. Prasad, A. C. Abhyankar, (2013) "*Annealing character of cartridge brass"*, MSAIJ, 9(3), pp-[97-106].

[4] R. Barron, (1982) "*Cryogenics"* 22 (5) pp-[409–413] Cryogenic Society of America.

[5] J. W. Kim, J. A. Griggs, J. D. Regan, R. A. Ellis, and Z. Cai, (2005) "*Effect of cryogenic treatment on nickel- titanium endodontic instruments"*. IntEndod J. 38(6), pp- [364–371].

[6] Kapoor, Jatinder, (2013) "*Effect of cryogenic treated brass wire on the performance of wire electrical discharge machining"*, Phd thesis presented to the faculty of engineering and technology of the Punjabi university, Patiala.

[7] I. D. Radomysel'skii, G. A. Baglyuk, & Mazharova., (1984) "*Soviet Powder Metallurgy and Metal Ceramics Volume 23*", Issue 3, pp-[56–64].

[8] Jery Tatiana Sabogal Vanegas, Mauricio Alejandro Sierra Cetina, (2016) "*Influence of the cryogenic treatment in micro structure, hardness and micro hardness of the brass 60-40 and bronze SAE*", International journal of engineering research & science Volume 2, Issue 8, ISSN 2395-6992.

[9] Raymond L. Patterson, (1945) New Jersey Zinc Co "*Powder metallurgy of brass*", US Patent No. 2368943A.

[10] Akhter H Ansari., M. Hameedullah & M S J Asghar, (2004) "*Stabilizing dimensions of brass powder components*", Indian journal of Engineering and Material sciences Vol. 11, pp-[481-486].

[11] R. Z. Vlasyuk, L. F. Kravchenko, V. D. Kurochkin, and I. D. Radomysel'skii, (1986) "*Evaporation of the Main Components of P/M brass During Heating*", UDC- [621-762].

[12] ASTM Standard B331, 1995, "*Standard Test Method for Compressibility of Metal Powders in Uniaxial Compaction*", ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

[13] ASTM Standard B925, 2003, "*Standard Practices for Production and Preparation of Powder Metallurgy (P/M) Test Specimens*", ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

[14] ASTM Standard B823, 2015, "*Standard Specification for Materials for Copper Base Powder Metallurgy (PM) Structural Parts*", ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.

[15] ASTM Standard E 92 – 17, 1997, "*Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials*", 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.