An Investigation into the Reliability of Some Locally Manufactured Crucibles and Possible Remedies.

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Abstract

Crucibles are important units in pyrometallurgical operations as they serve as containers within which high temperature reactions are conducted. For a given operation, good crucibles should be chemically inert, withstand high temperatures, thermal shock and prevent seepage of molten charge. In addition they should be robust enough to withstand the stresses involved in shipping and handling. Formerly, most of the crucibles utilized in the Ghanaian mining industry were imported but in recent times, some local crucible manufacturers have emerged. There are some challenges with the use of these locally manufactured crucibles as the failure rate is high, especially when used in fire assaying and in smelting of gold concentrates. In this study, the integrity of crucibles manufactured by two major local entrepreneurs, "A" and "B" was investigated. X-ray fluorescence spectroscopy indicated that the major minerals in the crucibles were Al_2O_3 and SiO_2 with crucible "A" having 20% and 42% respectively and 32% Loss On Ignition (LOI) while that in crucible "B" were 34% and 42.9% respectively with 17.5% LOI . Crucible "A" had feed particle size with 80% passing 78 µm while that of "B" was 54 µm. It was realised that failure was mainly due to the presence of coarse particles that reduce packing density and volatile material that escape on heating to create weak zones and channels that allow seepage of molten material. The combined effects of finer particle size, higher Al_2O_3 content and lower LOI gave crucible "B" superior performance over "A".

This paper critically investigated the credibility of local crucibles and presents recommendations.

The investigations included an audit of the processes involved, pre-smelting heating analysis, particle size analysis, volatile/combustible matter content analysis all in the vein of testing the crucible's chemical inertness, physical and mechanical strength etc.

Crucible preheating increased the success rate of the crucibles by about 20%. A good grind was necessary for good moulding of the crucible consequently strengthening it. Minimal combustible and volatile matter was advantageous.

Introduction

Refractory materials retain their strength at escalatedly high temperatures and should have negligible failures or change both physically and chemically. The chemical inertness and resistance to shock is the most favourable property of refractories. Kesse (1985) enumerated the uses and classified refractories as; alumina-silicates, high purity silicates, et cetera. The choice of refractories depends on the temperature, environment being worked in, the acidity or basicity of the slag, economic dynamics et cetera.

Crucibles are refractories that can withstand very high and arduous conditions during pyrometallurgical processes. They can be made from different materials such as graphite and certain types of clays, especially fireclay. Materials used in making crucibles should therefore be chemically inert during fusion. Good crucibles should withstand high temperatures without softening, withstand handling shipping and freighting, withstand thermal shock, be chemically inert and finally prevent seeping of molten charge and be impermeable to the products of combustion (Bugbee, 1940).

Geologically, Ghana has large deposits of refractory clays for the production of crucibles and other refractories; example, in Kumasi and Winneba, there are cumulatively about 2,544,923 and 64,545,195 metric tonnes respectively of clay (Kesse 1985).

In the suitable application of direct smelting (Styles et al, 2006) there is the need to use cheap, available and locally manufactured crucibles, mining companies and commercial metallurgical and analytical laboratories also have a high demand for crucibles in their operations. Previously, these industries were importing expensive graphite and dolomite crucibles.

For any country to be economically independent requires that they have minimal imports and maximize their export (Todaro 1982) not to even talk of the employment opportunities it presents. Currently, local entrepreneurship has directed its focus to this area and therefore the aforementioned industries now mostly use locally manufactured crucibles. The current study identified two locally manufactured crucibles from two different establishments in Ghana (Golvic Crucibles, Kumasi and Eckem Art and Pottery, Winneba) and one imported type Carmuese Lime Products, Inchaban). Eckem Art and Pottery makes about 600 crucibles per day and supplies about 7,000 to Intertek Laboratories, 'Societe General du Suveillance'

(SGS) Laboratories and Golden Star Resources Bogoso/Prestea Mine each per month. Golvic makes about 2000 unfired crucibles per day and about 35,000-40,000 crucibles per month, Golvic supplies many industries here in Ghana and the West African sub-region. This is proof that there is capacity for production.

Comparative tests in standard methods (Kesse 1985) were to ascertain the best quality and manufacturing procedures. These would help maintain a standard of production and thus sustain the industry.

Methods

Crucible Testing and Investigation

Several crucibles are available on the Ghanaian market, both imported and local. Types of crucibles available are presented in Fig 1.

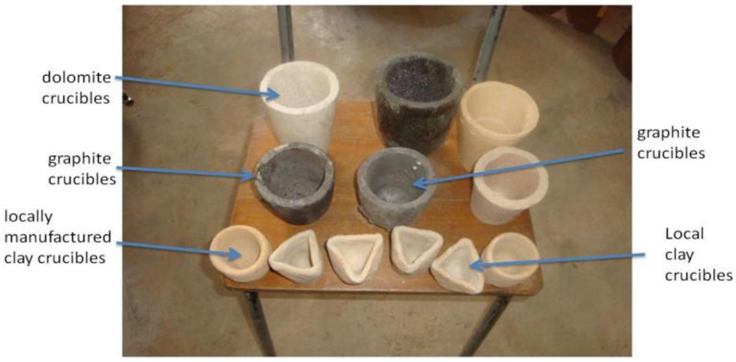


Fig.1. Different types of crucibles used in smelting operations

Manufacture Of Crucibles

For those manufactured in Ghana, the sequence includes mining of the clay followed by milling in a hammer mill or ball mill with porcelain balls as the grinding media. After mixing and moulding, the crucibles are sun dried for about three days and baked at about 1500^{o}C. Finally the crucibles are stored away from moisture and packed with saw dust, or shredded paper to prevent the formation of micro-cracks during loading and transportation (Figs 2-7).

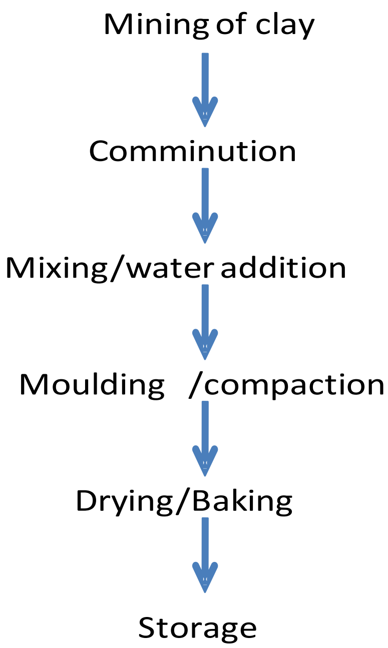


Fig.2. Process flowsheet for crucible manufacture

Fig.3. A hammer mill for milling clay

Fig.4 A crucible mould and press

Fig.5 Moulded crucibles being dried in the open

Fig.6 An oven for burning the crucibles

Fig.7 Manufactured crucibles in a storage facility

Crucible Testing

Crucibles from three major sources were tested to ascertain the particle size of materials used in the manufacturing process and the maximum temperature attained during calcinations. The effect of pre smelting heating and the slag composition during smelting on the performance and integrity of crucibles were also investigated. Thirty crucibles each were taken from the three manufacturers. For particle size analysis, two 100 g samples were taken from Eckem Art and Pottery and Golvic Crucible Manufacturers after grinding. Screening was done using a deck of test screens built according to the Tyler series from 250 µm to 45 µm for 15 minutes at 1 mm amplitude.

From interviews the crucible manufacturers indicated that calcination was conducted at 1500^{o}C. Two 100 g samples of fresh material were taken from Eckem Art and Pottery and Golvic Crucible Manufacturers and spread thinly on a ceramic platter and heated in the furnace at 1100^{0}C for twenty minutes, since gold melts at 1063^{o}C and smelting is usually done at about 1100^{o}C. The samples were then allowed to cool and weighed. One crucible each from each manufacturer was charged in a furnace in an attempt to simulate the arduous smelting conditions. The temperature was around 1150^{0}C, and their conditions after the runs were observed. To verify the effect of preheating the crucibles before charging; thirty more crucibles were taken from each manufacturer, preheated at 400^{o}C for up to 10 minutes before they were tested under the same conditions and their failure/success rates noted. Finally the three types of crucibles were prepared and submitted to the Ghana Geological Survey Department for X-ray diffraction (XRD) analysis.

Results and Discussions

Several crucibles were tested for competence. Some of the competence parameters that were investigated included high temperature stability, reactions with molten material, packing density and migration of molten material through the crucible walls. To establish these, some tests performed were the effect of preheating, particle size range of raw material, and volatile materials content. A test was adjudged successful if the smelting process goes to completion without a noticeable flaw in the crucible.

Effect of Preheating

Figure 8 shows the success rates of the various crucible manufacturers with and without preheating. Without preheating there were about 57%, 50% and 77% successes corresponding to Eckem, Carmuese and Golvic respectively. With preheating for 10 min at 400^{0}C, the success rates rose to about 80%, 67% and 97% also corresponding to Eckem, Carmuese, Golvic respectively.

A general increase in successes when the crucibles were preheated was observed for all three sources. This buttresses the fact that presence of moisture in the crucibles was a very major contributor to its failure. A study of temperature profiles during smelting (Abbey 2010), deduces that there is a rapid increase in temperature during smelting. This causes rapid expulsion of unbounded moisture leading to micro-cracks and the development of failure fault lines and zones. It is highly recommended that crucibles are preheated for about 10 minutes at a temperature of about 400^0C before charging.

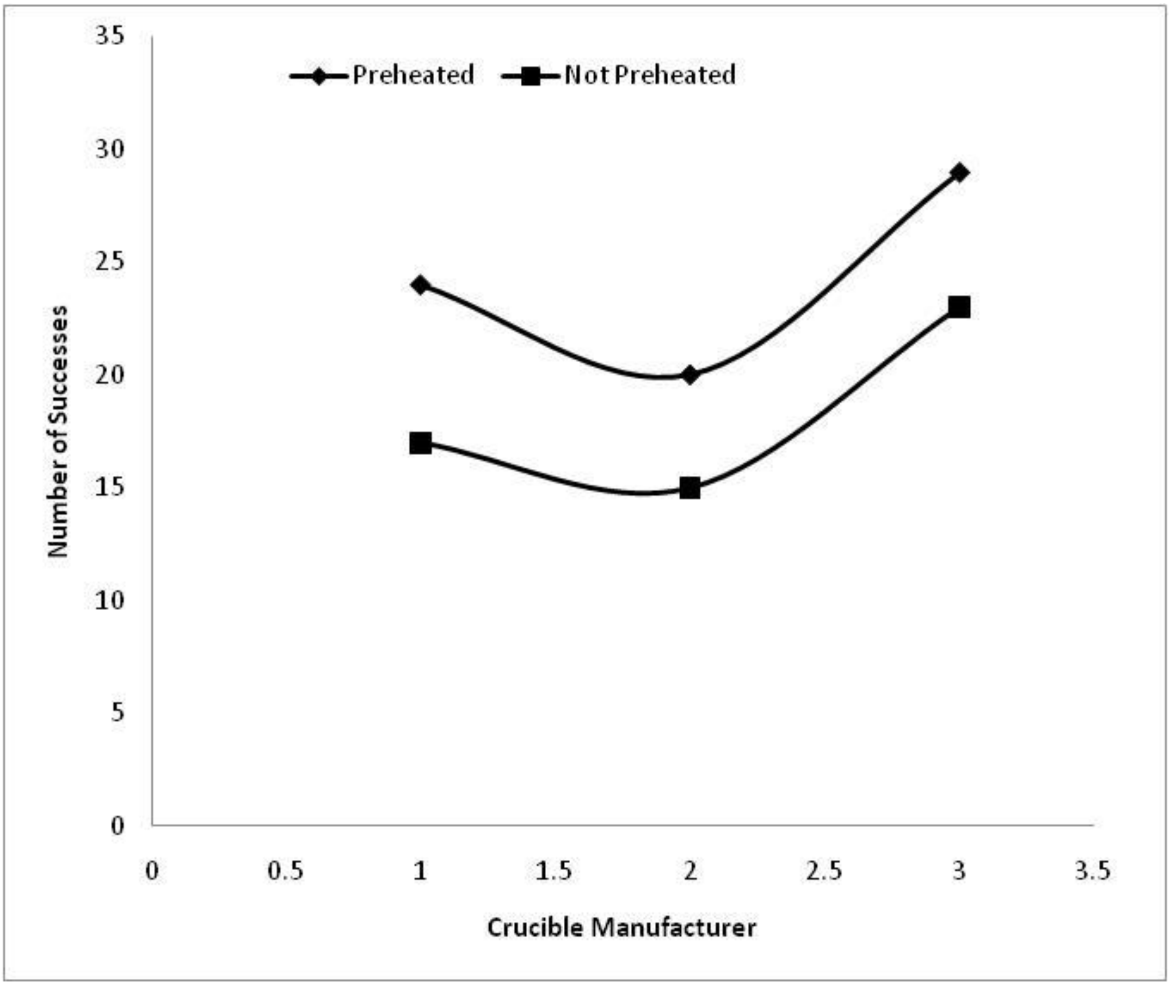


Figure 8 the success rates of the various crucible manufacturers with and without preheating.

Effect of Particle Size

Crucible manufacturers; as part of the preparation, grind the mined clay to a suitable particle size. Particle size has effect on packing density and hence migration of molten material through the crucible walls. As shown in Figure 9, the particle size of the products after grinding by the two manufacturers compared show that the P_{80} for Golvic and Eckem are about 54 and 78 microns respectively. Thus it is expected that when similar forces are applied during compaction, the Golvic material will have a higher packing density. Golvic had the highest success rate when the crucibles were tested, and all other parameters being equal, this is an indication that the finer grind used for moulding the crucibles conferred better performance. It is worth noting that Golvic uses a hammer mill while Eckem uses a ball mill for grinding with corundum balls as the grinding media. From initial appearance inspection Golvic crucibles had a better finish look. Since the crucibles from Carmuese Lime Products were imported it was difficult to get raw material for the particle size investigation and the volatile and combustible material investigation.

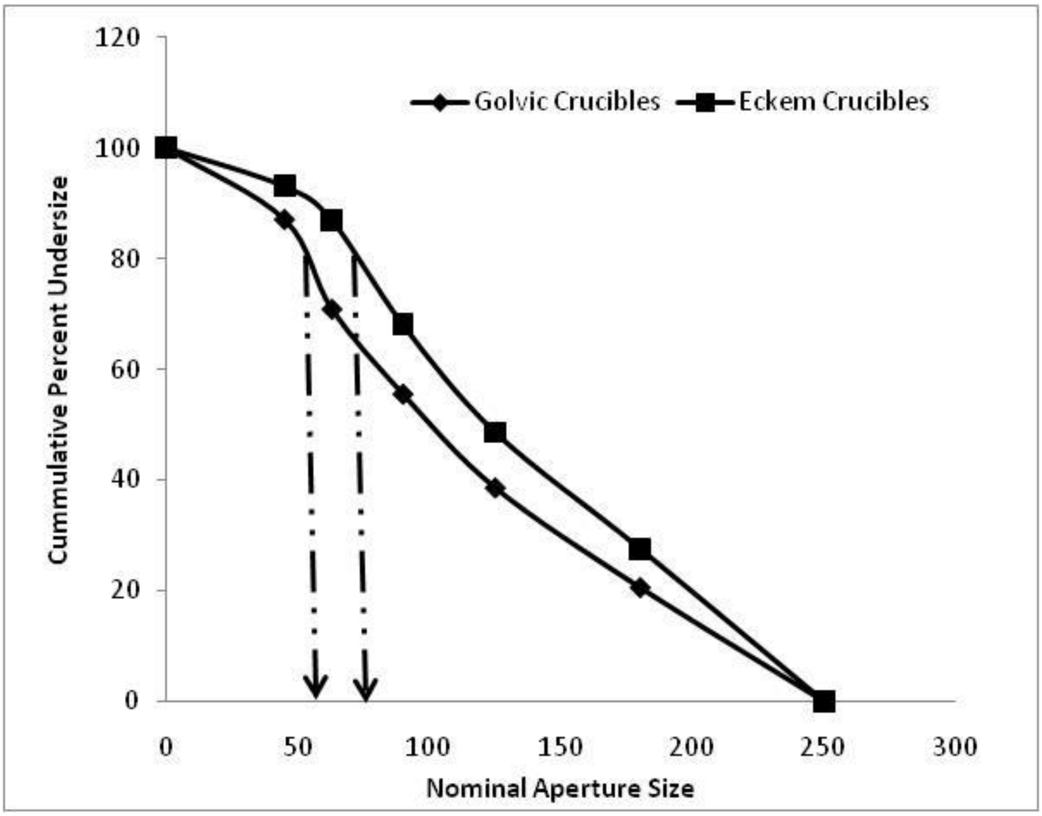


Fig. 9. The size distribution graph for the material.

Effect of Volatile and Combustible Material

This investigation was done to ascertain the effect of quantities of volatile/combustible matter in the clay used in manufacturing the crucibles, and to investigate a relation between the quantities of volatile/combustible material and crucible failure. From Figure 10 there was a reduction in weight of about 4 g and 17 g for Golvic and Eckem respectively when crucibles were heated for 20 min at 1100^0C. This meant that assuming crucibles made from the two manufacturers were charged under similar conditions, about 4% of Golvic crucibles and 17% of Eckem crucibles would be burnt out creating random voids that would allow molten charge to seep out (Figure 11). When the amount of seeped slag becomes excessive, the crucible soaks it up and at that temperature these conditions impart a soft and plastic nature and the crucible could even collapse under its own weight.

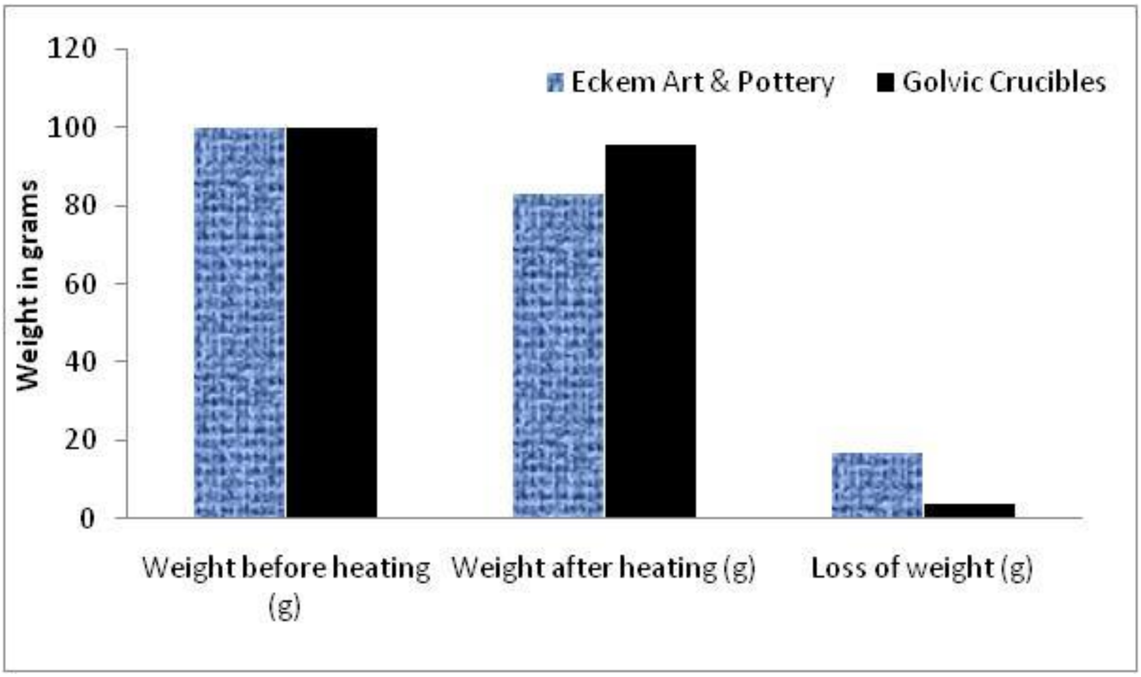


Fig. 10 Comparison of the two crucible makers before and after heating and their weight losses

Fig. 11 Molten charge that has seeped out of a crucible during fusion

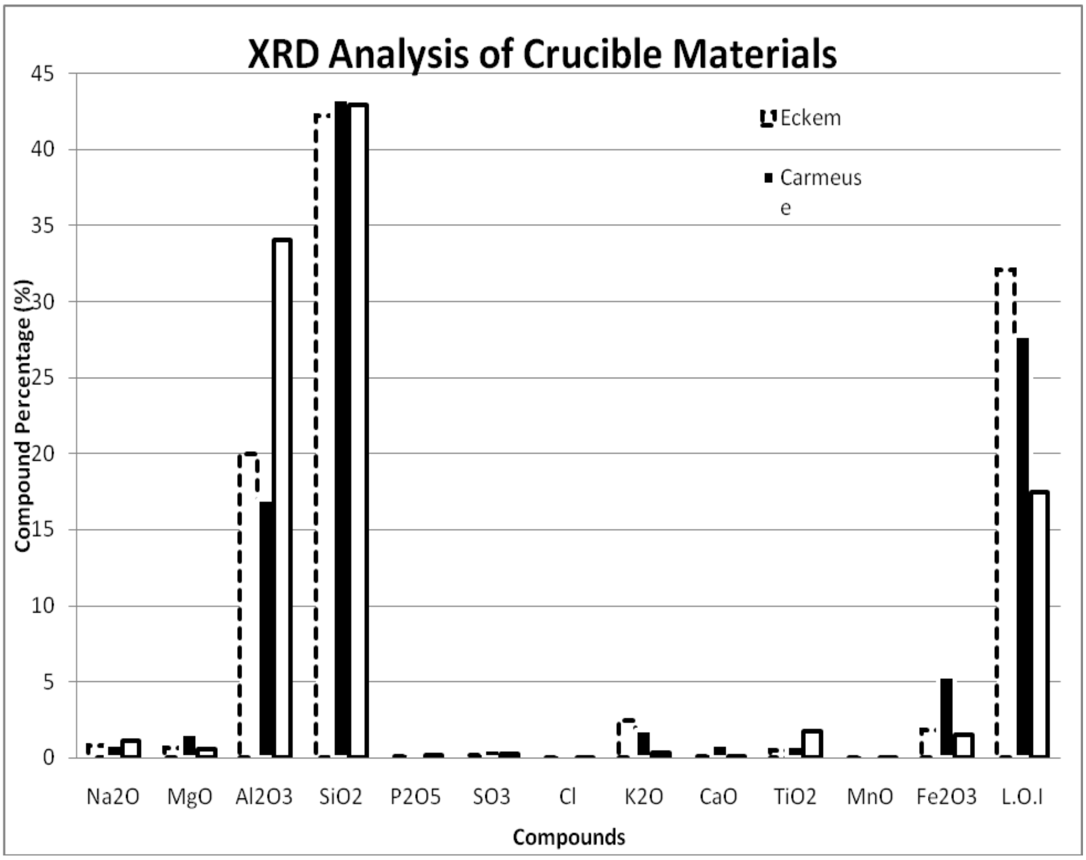
XRD Analysis

Major Oxides

Element	Dimension	Eckem	Carmeuse	Golvic
Na_2O	**%**	0.77	0.86	1.14
MgO	**%**	0.61	1.55	0.53
Al_2O_3	**%**	20.00	16.96	34.06
SiO_2	**%**	42.22	43.35	42.91
P_2O_5	**%**	0.06	0.10	0.19
SO_3	**%**	0.20	0.53	0.23
Cl	**%**	0.01	0.02	0.01
K_2O	**%**	2.45	1.80	0.34
CaO	**%**	0.05	0.91	0.08
TiO_2	**%**	0.45	0.78	1.71
MnO	**%**	0.01	0.05	0.01
Fe_2O_3	**%**	1.83	5.37	1.49
L.O.I	**%**	32.10	27.80	17.50
TOTAL	**%**	**100.76**	**100.08**	**100.19**

Alumino-Silicates are known to be very good refractory materials, the aim was to establish the alumina and silicate quantities of the various materials. Golvic had the highest Alumina content then Eckem and finally Carmeuse; this could explain their performances. The silicate quantities were however comparable.

Loss On Ignition (LOI) was also lowest for Golvic, followed by Carmeuse and then finally Eckem. This confirms the earlier fact that the amounts of volatile/combustible matter was proportional to risk of failure.



Conclusions

Preheating of the crucible increased the success rate of the crucibles by about 20%. A good grind was necessary for good moulding of the crucible to give it good strength. Minimal combustible and volatile matter also prevents seepage of slag which consequently causes crucible failure. The XRD analysis totally confirms the previous discussions.

Recommendations

Manufacturers

Crucible manufacturers should have very fine grind to allow for good packing of particles in the crucible, this prevents seepage. Volatile and combustible materials must be reduced to the barest minimum. When packing for freighting, transportation; there should be sufficient stuffing to reduce shocks and micro-crack formations. When crucibles are to be stored for time they should be in arid and dry places.

Consumers

Consumers could preheat crucibles for about 10 minutes at a temperature of about 400^0C before use. Storage should be done in dry sheds where rain or humid conditions are minimal.

References

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