EFFECT OF FIRRING HEATING RATE ON THE DENSITY, POROSITY, VICKERS HARDNESS AND MICROSTRUCTURE OF THE CRUCIBLE SPECIMENS

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Abstract

Failure in the results of making the crucible is cracking and even deformation. Cracks that occur can cause fluid leakage when melting so that it disrupts the casting process. The quality of the crucible can be influenced by factors including the selection of constituent materials, particle size, the amount of pressure, and the length of the firring process of heat treatment and cooling in the furnace. This study aims to determine the effect of heating rate on crucible made from clay and kaolin and molasses as a binder. With the treatment of different heating rates in the firring process, namely 3 ℃ / min, 4 ℃ / min, 5 ℃ / min, 6 ℃ / min, and 7 ℃ / min. The composition of the materials used is 40% clay, 40% kaolin, and 5% molasses and 15% water as the total mass. The process of making specimens begins with crushing clay, sieving clay 100 mesh. Mixing process using twin screw extruder machine repeatedly for 45 minutes. Molded cylindrical size 20 x 20 mm, free air drying for 8 days, firring process at 1000 ℃ holding time 1 hour. The results showed that the difference in heating rate did not affect the change in chemical elements but the best heating rate was at 3℃/min showing a denser morphology, density value of 1.62 g/cm^3, porosity value of 23%, and Vickers hardness value of 20.43 HVN.

***Keywords**: Crucible, Heating Rate, Physical Properties, Mechanical Properties*

INTRODUCTIONS

The global ceramic vessel market was valued at USD 1271.06 million in 2021 and is expected to reach USD 1418.71 million by 2027 (Business Research Insights, 2024). However, in some cases failures occur when the crucible is used for the metal melting process (Rusiyanto *et al.*, 2022). Failure in the results of making the crucible is cracking and even deformation. Cracks that occur can cause liquid leakage when melting, thus disrupting the casting process, namely shrinking the volume of the melting liquid. A decrease in temperature causes casting failure because it does not produce the desired casting product. Meanwhile, deformation is caused by the crucible material which cannot withstand high temperatures during the melting process. Crucible is a vessel that has a higher melting point than the material to be melted (Hendronursito *et al.*, 2019). The crucible serves as a physical barrier that separates the heat source from the molten metal, hence playing a crucial role in determining the efficiency of metal melting (Pinto *et al.*, 2019). The critical determinants of heat transfer through a crucible are thermal conductivity, specific heat capacity, and shape, which are fixed properties. The heat resistance of crucibles is essential for their efficacy in several industrial and scientific procedures that entail the fusion and molding of metals. The crucible material must possess elevated melting temperatures and be capable of enduring and confining the molten metal (Fashu *et al.*, 2020).

A crucible is a container made of ceramic or metal that is used to melt or refine metals and their alloys at very high temperatures (Przylucki *et al.*, 2018). The crucible or container can be used to melt non-ferrous alloys, copper, aluminum, and tin. Cast iron melting can be done using a low-frequency induction furnace. High-frequency induction kitchens are used to melt materials that are resistant to high rates. Small and medium-sized aluminum smelters usually use crucible furnaces. The induction heating system consists of a water-cooled copper

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induction coil, a graphite crucible, and a thermal refactory (Rusiyanto *et al.*, 2022).

Crucible must have physical properties with high purity, fine grain, and good heat conductivity, high density, low porosity, thermal shock resistance, corrosion resistance, good thermal stability, high mechanical strength, low permeability and good oxidation resistance (Chen *et al.*, 2019). The quality of the crucible can be influenced by factors including the selection of constituent materials, particle size, the amount of pressure, and the length of the firring process of heat treatment and cooling in the furnace. The heating rate is the increase in temperature every minute in the firring process. In making crucible, it is necessary to know the parameters of the right heating rate because it will determine the length of time in the crucible firring process so that it will affect the crucible results. The use of crucible is also related to temperature and to achieve the desired temperature there must be a heating rate at each temperature and minute until each material blends perfectly (Habiby *et al.*, 2022).

Microstructural changes occur during firing when sintering takes place, the pores change size and shape (Callister, 2007). Firing clay too quickly leads to crumbling and cracking. This is due to insufficient time for water to evaporate (Sulistya, 2016). Increasing the heating rate increases porosity, decreases density, compressive strength drops significantly in lightweight foam ceramics (Chen *et al.*, 2021).

The firing process converts raw clay into ceramics by subjecting it to high-temperature heating (Hamid *et al.*, 2021; BBC Bitesize, 2024). Modifications in the temperature and duration of fire significantly impact the quality of bricks. Lowering the temperature at which the firing process occurs and reducing the duration of the firing process not only decreases the expenses associated with manufacturing but also enhances the efficiency of the plant (Karaman *et al.*, 2006). Miras et al. studied the mineralogical changes of two clay materials with different compositions during burning using XRD and HTXRD methods (Miras *et al.*, 2018). Csákia et al. examined the electrical conductivity of alternating current (AC) on illite clays during the firing process to identify the primary mechanism responsible for the conductivity (Csáki *et al.*, 2018). Štubňaa et al. conducted a study on the phase development and its impact on these clays' elastic and inelastic mechanical properties during combustion's heating and cooling stages (Štubňa *et al.*, 2018).

Heating rates refer to the pace at which the temperature increases during combustion, and they play a crucial role in determining the qualities of the crucible material (Habiby *et al.*, 2022). The rate of heating throughout the firing process is critical to determining the ultimate qualities of the product. The rapid fire process leads to the expansion of clay due to the creation of a non-porous vitrified exterior layer, which hinders the escape of gases like water vapour and CO_2 from the clay's interior (Karaman *et al.*, 2006). In this study the authors focused on the treatment of heating rate in the firing process of crucible. In this study it is expected to determine the heating rate that is good in terms of physical properties and mechanical properties in the crucible firring process. This study aims to determine the physical properties, namely density, porosity and make observations with a Scanning Electron Microscope-Energy Dispersive X-Ray or SEM-EDX to observe the morphology and composition and mechanical properties of Vickers hardness after being treated with different heating rates in the firing process.

MATERIALS AND METHODS

The parameters of this study use independent variables, namely the heating rate of 3°C/min, 4°C/min, 5°C/min, 6°C/min, and 7°C/min (Li *et al.*, 2020; Fu *et al.*, 2023), related variables are density, porosity, Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX), and Vickers hardness. Control variables are sieving clay 100 mesh (Siswoyo *et al.*, 2023), composition of clay 40%, kaolin 40%, molasses 5%, and 15% water as the total mass, mixing using a twin-screw extruder machine repeatedly for 45 minutes (Akbar *et al.*, 2022), molding using mold claims without compaction pressure, drying specimens for 8 days at room temperature, firring with a temperature of 1000 °C held for 1 hour (Amuda *et al.*, 2019; Hidayat *et al.*, 2022). This research uses a quantitative approach method. Research with a quantitative approach emphasizes the use of numbers starting from data collection, interpretation of data and presentation of results. Presentation of results in the form of pictures, tables, graphs or other displays that facilitate the delivery of information so that readers can understand.

This research uses tools and materials, namely a drilling machine complete with cutting edges to pulverize clay into powder, 100 mesh sieve, digital scales, hammers, used putty cans, cable rollers, used gallons, twin screw extruder machines, claim molds, nabertherm industrial furnaces, sandpaper, calipers, plastic clips, digital balance, aquadest, vaseline, rags, microhardness tester M800, hacksaw, Phenom/Pro for SEM/EDX testing. While the materials used are clay, kaolin, molasses, and water.

Making specimens starts from crushing clay into powder and then sieving 100 mesh, mixing is done using a twin-screw extruder machine for 45 minutes repeatedly, specimens are molded using mold claims without compaction pressure, then dried for 8 days at room temperature before the firring process.

RESULT AND DISCUSSIONS

The density test results show that the difference in heating rate treatment variations from a slow heating rate of 3°C/min to a fast-heating rate of 7°C/min causes a decrease in density, but the decrease is not too significant (Figure 1).

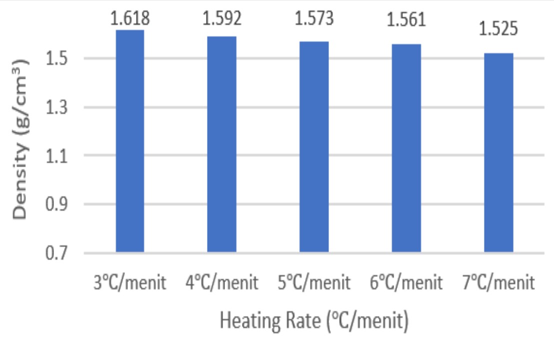

Figure 1. The results of density testing

This shows that the crucible specimen with the slowest heating rate has a better density than the crucible specimen with a fast-heating rate. Relevant research results have also been carried out by Amuda et al who conducted research on the manufacture of refractories from synthetic alumina / silica mixtures with properties close to standard alumina / silica for refractory purposes (Amuda *et al.*, 2019). The linear shrinkage of the refractory blends was within the permissible limits of 0-4%, specific gravity of 1.55-1.89 g/cm3, water absorption of 21.7-30.4% and compressive strength in the range of 1,125-2,017 KN/M2. The apparent porosity at 45% is outside the standard for fireclay refractory materials.

The porosity test results show that the difference in heating rate treatment variations from a fast-heating rate of 7°C/min to a slow heating rate of 3°C/min causes a decrease in porosity, but the decrease is not too significant (Figure 2). This shows that the crucible specimen with the slowest heating rate has better porosity than the crucible specimen with a fast-heating rate. Relevant research results have also been conducted by [15] with heating rates of 2 ºC/min, 4 ˚C/min, 6 ºC/min and 8 ˚C/min and a sintering temperature of 700˚C/min. The slowest heating rate produces good physical properties of glass ceramic composites (Salleh *et al.*, 2017).

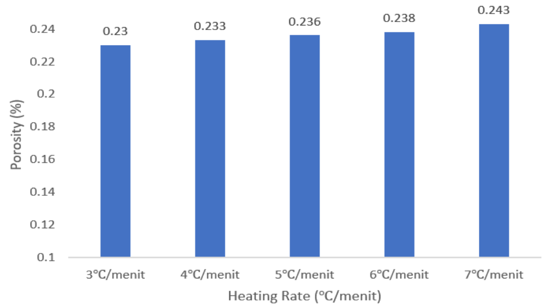

Figure 2. The results of porosity testing

The results of the Vickers hardness test show that the crucible specimens with a slow heating rate of 3°C/min to a slow heating rate of 7°C/min decrease the Vickers hardness (Figure 3). This shows that the slow heating rate of 3°C/min is a good treatment for burning bowls to get a good hardness value compared to the fast-heating rate of 7°C/min in burning bowls. Relevant research results have also been conducted by [10] at a heating rate of 0.5 °C / min to 5 °C / min the Vickers hardness of alumina ceramics decreases from 266.5 HVN to 78 HVN. This happens as the heating rate increases.

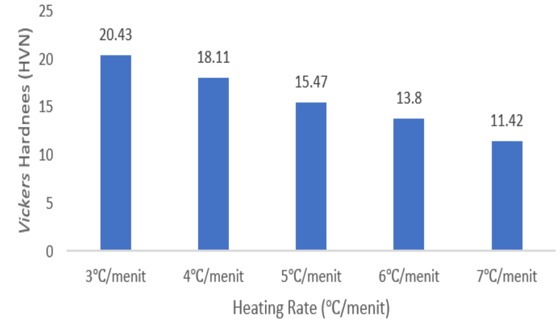

Figure 3. The results of hardness testing

The EDX test results of chemical element composition show that oxygen, silicon, and aluminum are evenly distributed. These results indicate that both specimens treated at 3°C/min (Figure 4) and 7°C/min (Figure 5) have the same chemical elements. Thus, the heating rate has no effect on changes in chemical elements. At 3°C/min shows that the element O (Oxygen) with a percentage of 58.82%, Si (silicon) with a percentage of 15.39%, Al (Aluminum) with a percentage of 11.66%, and N (Nitrogen) percentage of 12.55%. Whereas in the EDX test results of 7 °C / min treatment there are several elements that dominate the most in percentage, namely Si (silicon), Al (Aluminum), and N (Nitrogen). At 7°C/min shows that the element O (Oxygen) with a percentage of 69.46%, Si (silicon) with a percentage of 10.7%, Al (Aluminum) with a percentage of 8.24%, and N (Nitrogen) percentage of 10.52%. These results show that both 3°C/min and 7°C/min treated specimens have consistent chemical elements with different atomic percentages. Thus, the heating rate has no effect on changes in chemical elements.

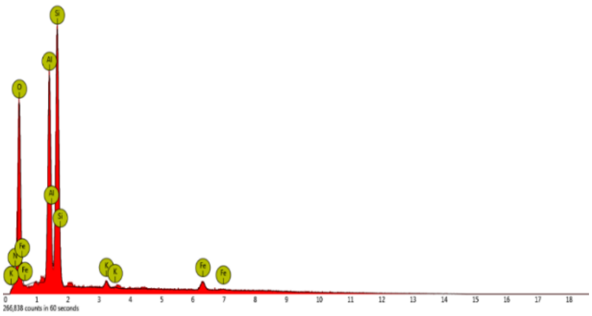

Figure 4. EDX testing on the 3°C/min specimens

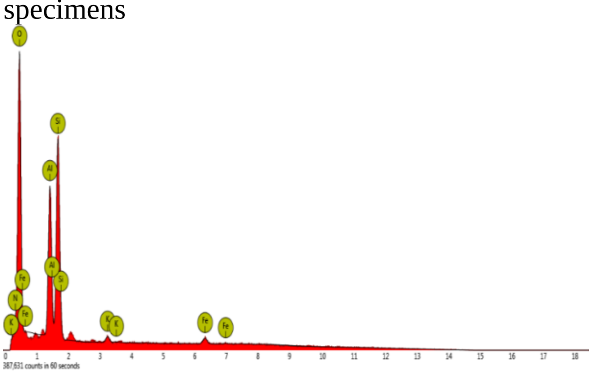

Figure 5. EDX testing on the 7°C/min specimens

4. Conclusions

The research data obtained shows that a slow heating rate of 3°C/min is very good at increasing density, Vickers hardness, and a tight microstructure and reducing the porosity of crucible material compared to a fast-heating rate of 7°C/min. Because the slow heating rate will make the process of burning the material in the furnace slowly to reach the sinter point so that the process of movement between the grains of the material occurs slowly and can fill the voids between the grains in the crucible material better, so it can be concluded that the use of a slow heating rate can improve the physical properties and mechanical properties of the crucible material.

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