

Original Article

Analysis of surface blistering in the manufacturing of faucets*

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Received: 10 September 2023; Revised: 15 December 2023; Accepted: 10 January 2024

Abstract

This research aimed to carry out an analysis of the surface blistering problem in the manufacture of faucets. Zamak 3 is a material produced by high-pressure die-casting. The surface blistering microstructure was examined with an optical microscope and a scanning electron microscope, while gas properties were examined by using gas chromatography. The results from optical and scanning electron microscopy showed discontinuities in the zinc layer, including a large amount of porosity spreading all over the product on the surface of the zinc layer as well as blistering under the surface. The gas chromatography showed that the average amount of hydrogen gas was 0.00755 percent, supporting the hypothesis that the discontinuities are caused by the porosity containing gas inside the base metal, and the heat during liquid metal injection is the main factor driving gas expansion, affecting the discontinuities.

Keywords: surface blistering, faucet, Zamak 3, high pressure die-casting (HPDC), gas chromatography

1. Introduction

Zamak 3 is the most common Zamak alloy and a popular material for die-casting (Kaye & Street, 2016). Zamak comprises a family of alloys with zinc as the base metal and aluminum, magnesium, and copper as alloy elements. Zamak is widely used in the production of automotive parts, furniture accessories, appliance components, faucets and taps, shower hinges, handles, valves and pneumatics, toys, ornaments, clothing, and mechanical parts, as well as in other industries and applications (Campbell, 2015; Schweitzer, 2003; Wang, Turnley, & Savage, 2011). Different Zamak alloys are suitable for different uses. When it comes to Zamak 3 alloy, it is usually considered the first choice for zinc die-casting (Zhao, Wang, Li, & Xia, 2009). The high-pressure die-casting process uses high pressure to inject molten metal into the

mold cavity until it is filled by casting in a hot chamber (Zhao, Wang, & Xia, 2009). It is commonly used with alloys that have a low melting point, such as the zinc alloy that has a melting temperature of approximately 420 degrees Celsius. The outstanding feature of the hot furnace metal casting machine is that the temperature of the molten metal is maintained during injection, which shortens the casting time and increases productivity (Campbell, 2015).

Surface blistering is a form of defect that occurs in high-pressure die-casting. It is caused by the entrainment of gases within each sub-surface layer of the metal when metal is injected into the mold (Armillotta, Fasoli, & Guarinoni, 2016). Panossian *et al.* (Panossian & Ferrari, 2004) studied blistering problems in zinc-plated steel and zinc-plated die-cast zinc alloy parts. The results revealed that blistering was caused by a variety of factors. For zinc-plated steel parts, it was caused by inadequate pre-treatment, and for zinc-plated die-cast zinc alloy parts, it was caused by poor substrate surface quality.

Oksana *et al.* (Ozhoga-Maslovskaja, Gariboldi, & Lemke, 2016) studied the conditions for blister formation during thermal cycles of Al-Si-Cu-Fe alloys for high pressure die-casting. The results indicate that blisters developed from

*Peer-reviewed paper selected from the 10th International Conference on Engineering and Technology

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pores below a critical aspect ratio, as a result of high plastic strain accumulation in localized regions lying between the pore and outer surface (ligament).

Reveko (Reveko & Moller, 2018) studied the special aspects of electrodeposition on zinc die casting, for which zinc and aluminum are well-known chemically active elements, especially in the molten state. These metals can react with moisture and other substances present in the mold, leading to gas formation. As a reaction product, hydrogen can be easily dissolved in the molten metal. Subsequently, molecular hydrogen together with some trapped air form bubbles when the melt starts to solidify, which remain in the bulk since the outer solid skin is already formed, resulting in inner porosity.

In this study, an analysis of surface blistering on the faucet resulting from high-pressure die-casting was conducted to determine its root cause. The surface blistering and the nearby microstructure were observed and analyzed using optical microscopy (OM) and scanning electron microscopy (SEM). Gas chromatography was employed to inspect the entrained gases within a sub-surface layer of the metal. The interpretation of the study's results facilitated the provision of recommendations for mitigating surface blistering defects in future manufacturing processes.

2. Materials and Methods

In this research, Zamak 3 was utilized as the material for the faucets. The chemical composition of the faucets was analyzed using an optical emission spectrometer (OES) (Hitachi, model OE750). The specific chemical composition of the faucets is presented in Table 1.

Table 1. Chemical composition of faucet

Element	Zn	Al	Cu	Fe	Mg	Si
Weight (%)	94.49	4.72	0.574	0.0628	0.0436	0.011

Three metallographic samples (B1, B2, and B3) exhibiting surface blistering were sectioned using a cut-off wheel. The metallographic specimens were then polished with silicon carbide paper and diamond paste, followed by etching with a solution of 5 ml nitric acid in 100 mL of ethanol. The swab technique was applied for 5 seconds during etching. Post-etching, the samples were thoroughly washed with flowing water and alcohol, and then dried using hot air.

The metallographic examination was conducted using optical microscopy (OM) with a Nikon model Eclipse Nvdi-N light microscope. Microscopy was complemented by chemical microanalysis using energy dispersive spectrometry (EDS) and scanning electron microscopy (SEM) to characterize the precipitate phases. The SEM tests were performed with a JEOL model JSM-6610LV.

The gas analysis was conducted using gas chromatography with the GC 7890A device equipped with a thermal conductivity detector (TCD). The procedure involved: 1) selecting four samples from the faucet body with blistering issues for testing; 2) preparing test pieces by cutting them into small sizes suitable for vials, and placing them into four tubes (including one tube without a test piece for comparison); 3)

weighing each workpiece in the vials and recording the values for calculations, followed by placing the vials in an oven at 80 degrees Celsius for 30 minutes; 4) extracting the gas inside the vial using a syringe; 5) calculating the weight fraction of hydrogen gas to facilitate a comparison of gas amounts in each vial using Equation (1), as illustrated in Figure 1 (a-d).

$$\text{Hydrogen per weight ratio} = \frac{\text{Hydrogen (gram)}}{\text{Weight (gram)}} \quad (1)$$

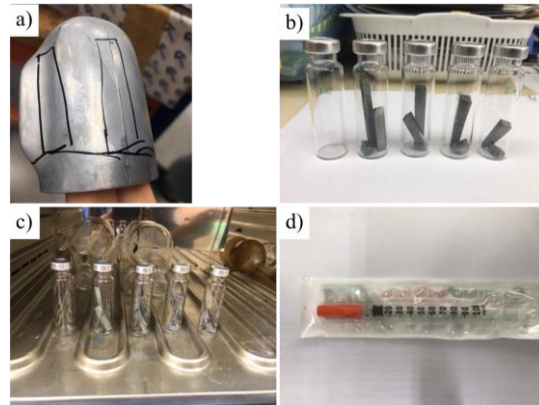


Figure 1. Gas chromatography set-up

3. Results and Discussion

3.1 Visual examination

The results of the macroscopic observations performed on specimens B1, B2, and B3 found surface blistering with a size of around 1 mm, after electroplating and polishing to make the surface shiny and have the desired properties. There were 3 layers of coating: copper, nickel, and chromium. The surface blistering is shown in Figure 2.

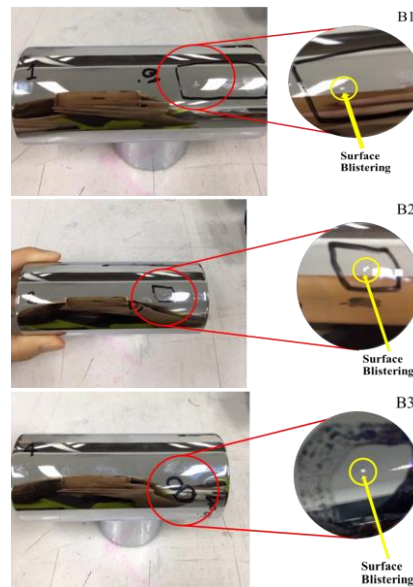


Figure 2. Results of visual examination showing the surface blistering on specimens B1, B2, and B3

Surface blistering in zinc die-casting typically occurs due to the entrapment of gases, often nitrogen or hydrogen, within the molten zinc during die-casting. These gases can get trapped as the metal solidifies, resulting in the formation of surface blisters (Pola, Tocci, & Goodwin, 2020; Sakuragi, Fuwa, Nakayama, & Nagamori, 2020).

In the case of surface blistering on a faucet, the process involves compressing the air within a high-pressure die-cast machine and ejecting the component from the mold using mechanisms such as a vacuum valve, overflow system, or vent. Any residual air present inside the casting dissolves uniformly into the molten metal. The turbulence within the process can lead to localized air concentrations, resulting in high-pressure cavities. After electroplating the faucet also undergoes heating, causing any accumulated air beneath the applied coating to escape. This release of trapped air can lead to blisters on the faucet surface (Dybowski, Kielbus, & Poloczek, 2023).

3.2 Structural characterization by optical microscopy and scanning electron microscopy

The typical microstructure of specimens in Figures 3 (a, c, and e) illustrates that the chill zone of a workpiece features fine grains. Deeper within the material, dendritic grains are evident in the columnar zone. As we progress towards the center of the workpiece, known as the equiaxed

zone, the grains maintain a round shape similar to those near the surface, though larger in size (Ares, & Gassa, 2012; Ares, & Schvezov, 2007; Zhang, Shortle *et al.*, 2019).

Figures 3 (b, d, and f) reveal a coating consisting of two layers. The first layer is copper, with thicknesses of 21.72, 19.27, and 23.00 micrometers, respectively. On top of this layer lies another composed of nickel and chromium, measuring 6.42, 5.27, and 5.65 micrometers in thickness, respectively. Additionally, there is a discontinuity layer, situated beneath the copper layer, also having copper inserts into the discontinuity area and cracks at the Ni and Cr layers. The crack arises from localized plastic strains occurring at different temperatures during the electroplating (Ozhoga-Maslovskaja, Gariboldi, & Lemke, 2016).

Figure 4 illustrates the detailed microstructure of the as-annealed sample, characterized by back-scattering electrons (BSE). As depicted in Figure 4 (a), the microstructure reveals the presence of η -Zn, while Figure 4 (b) highlights the eutectic phase consisting of fine Zn and Al phases (Hasan, Sharif, & Gafur, 2020; Liang, Wu, Sandl obes, Korte-Kerzel, & Schmid-Fetzer, 2019). The Zn matrix is observed to contain fine α -Al phases. Al particles with varying sizes form under different conditions: specifically, the eutectic-phase Al particles crystallize at high temperatures, while ultrafine-grained Al particles precipitate in the Zn matrix during cooling that decreases solubility (Pola, Tocci, & Goodwin, 2020).

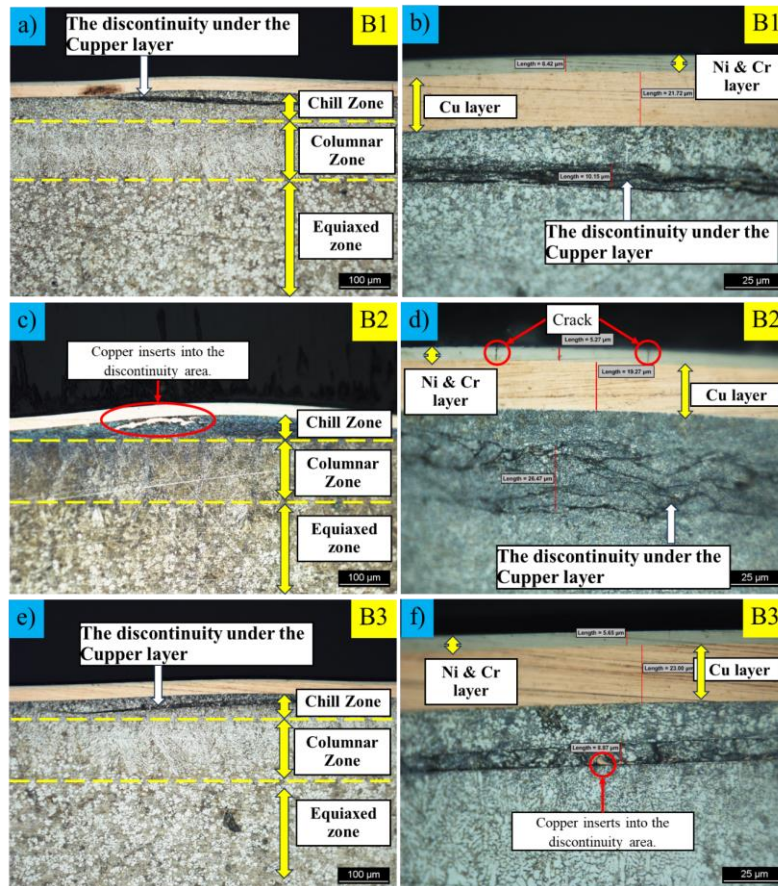


Figure 3. Cross-sections of blistered specimens B1, B2, and B3

Figure 5 displays the SEM-EDS results of the base material (specimen B1). Upon closer examination, several distinct phases were identified through point analysis. The Spectrum 1 area is η -Zn-rich, characterized by round, white regions distributed along the grain boundaries. Spectrum 2, analyzed over an area, displays a eutectic structure consisting of the two phases η -Zn and α -Al. Quantitative elemental analysis techniques were employed to determine the compositions of these phases: 1) The η -Zn phase (Pola, Tocci, & Goodwin, 2020) is identified as a phase with a substantial amount of zinc dissolved in the form of a solid solution, constituting a zinc-rich solid solution. This phase has a higher quantity of zinc than what is proportionally expected based on the total weight of zinc in the workpiece. 2) Conversely, the α -Al phase contains a relatively high concentration of aluminum dissolved in the form of a solid solution mixed with zinc (Pola, Tocci, & Goodwin, 2020). It was also observed that the amount of aluminum element in this phase was higher than what would be expected proportionally based on the total weight of the aluminum element in the workpiece (Pola, Tocci, & Goodwin, 2020).

The presence of carbon suggests an active role in contamination within the microstructure. The quantitatively measured carbon by Energy Dispersive X-ray Spectroscopy

(EDS) analysis may be attributed to the die lubricant agents on the mold surface. It has been reported that such reactions occur during die-casting. For example, if the lubricant agent is octadecane, the reaction would be $C_{18}H_{38} = 18C + 19H_2$. This reaction illustrates the generation of carbon and molecular hydrogen. Carbon remains effective as a barrier for the melt, and the hydrogen contributes to the development of internal porosity (Reveko & Moller, 2018).

The microstructures of specimens B1, B2, and B3 were examined using scanning electron microscopy, and the results are presented in Figure 6. In all three specimens, discontinuities were identified beneath the coating surface. Figures 6 (a), (c), and (e) are images obtained through backscattered electron imaging (BEI), which enhances the contrast between elements with different atomic numbers, allowing for a clear visualization of the grain characteristics in each structure. On the other hand, Figures 6 (b), (d), and (f) display secondary electron images (SEI). These provide depth-of-field information, enabling the identification of areas where discontinuities, such as hollow regions, are present, making it possible to analyze the elemental composition of both the bulging surface and the discontinuities within the blistering surface.

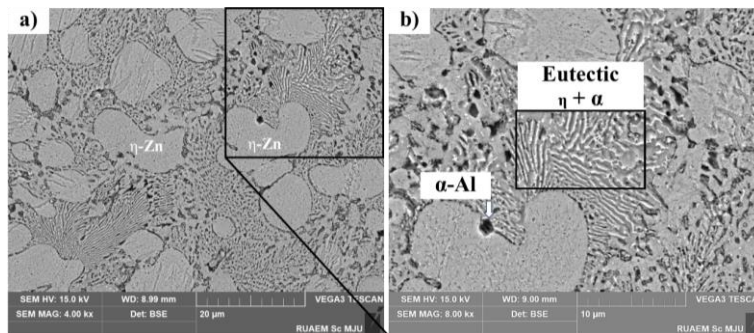


Figure 4. Typical morphology of the base material (specimen B1): (a) η -Zn phase, and (b) α -Al particles and the eutectic lamellar morphology ($\eta + \alpha$)

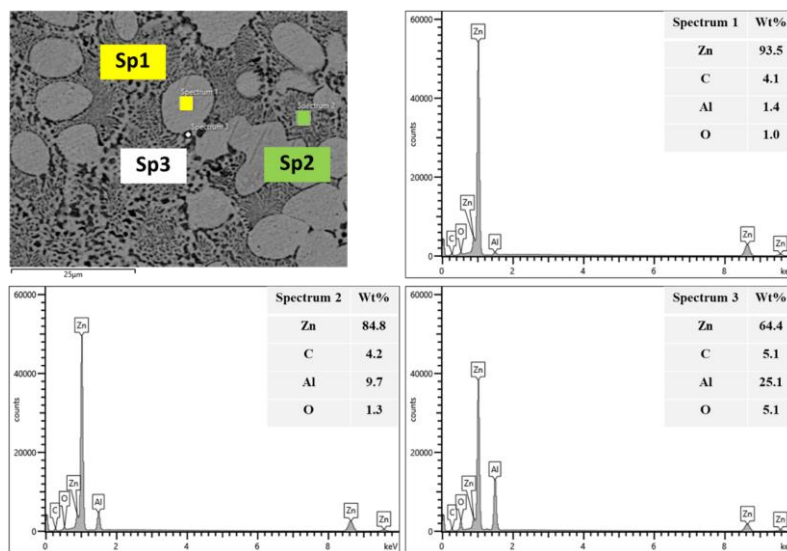


Figure 5. SEM-EDS results for the base material (specimen B1)

Figure 7 reveals the elemental content of the workpiece by applying a line scan at the surface blistering, which found that the amount of zinc decreased suddenly in the area of the blistering surface, indicating that the material had disappeared from that area and a gap had formed. Elemental analysis using linear scan also confirmed that the coating had three layers comprised of chromium, nickel, and copper, with the respective layers having thicknesses of approximately 3 micrometers, 6 micrometers, and 21 micrometers.

3.3 Results of gas inspection using gas chromatography

The gas inspection results for each sample obtained from gas chromatography are presented in Table 2.

Table 2 displays the hydrogen gas concentrations found in four specimens. Specimen 1 exhibited a hydrogen gas concentration of 0.1537 percent, while Specimen 2 had a hydrogen gas concentration of 0.1213 percent, Specimen 3 had a hydrogen gas concentration of 0.1313 percent, and the fourth specimen contained 0.1282 percent hydrogen gas. Since the specimens varied in size and weight, it was necessary to calculate the proportion of hydrogen gas to weight for comparison. Specimen 1 had a hydrogen gas concentration of 0.0083 percent per gram, while Specimen 2 had 0.0072 percent per gram of hydrogen gas, Specimen 3 had 0.0071 percent per gram of hydrogen gas, and Specimen 4 had 0.0076 percent per gram of hydrogen gas.

In zinc die casting, the blistering can occur due to various factors including the electroplating process. Blistering

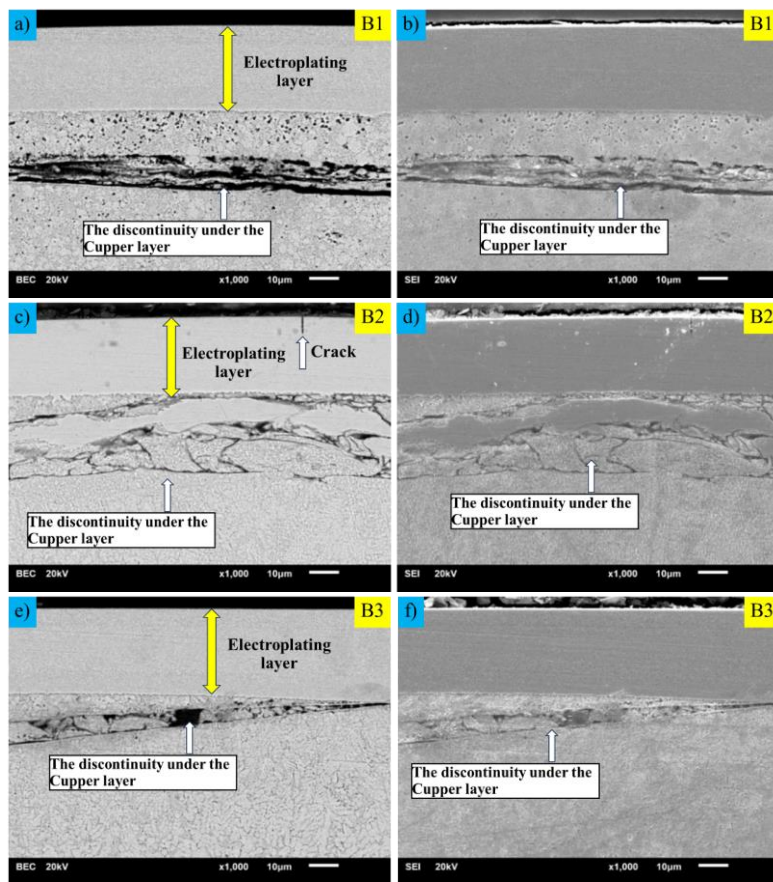


Figure 6. SEM (BEC and SEI) results for the specimens B1, B2, and B3

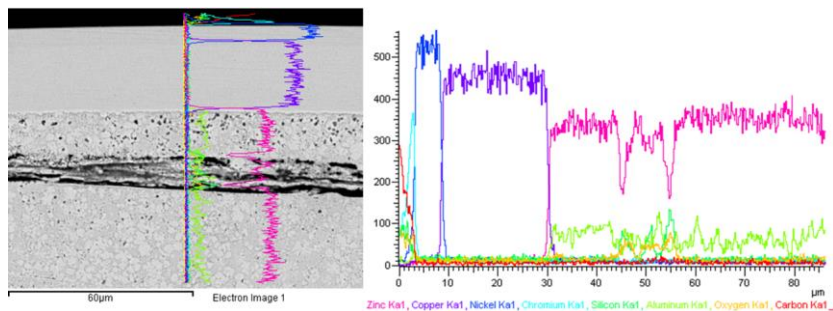


Figure 7. Graph showing the proportions of elements in the surface blistering area

Table 2. Amounts of hydrogen gas found in the workpieces using gas chromatography

Compound name	Results (%Norm)			
	Sample 1	Sample 2	Sample 3	Sample 4
Sample weight (grams)	18.5709	16.9007	18.6004	16.7804
Hydrogen	0.1537	0.1213	0.1313	0.1282
%Hydrogen per weight (%/g)	0.0083	0.0072	0.0071	0.0076
Average %Hydrogen per weight (%/g)	0.00755			

involves the formation of bubbles or blisters on the surface of the zinc die-cast part after electroplating. It is often considered a defect that can impact the appearance and functionality of the final product. For specimens B1, B2, and B3, surface blistering was detected after the electroplating.

The electroplating can contribute to blistering in zinc die-casting (Panossian & Ferrari, 2004; Reveko & Moller, 2018) due to contamination, in which contaminants on the surface of the zinc die-cast part, such as oils, greases, or residual mold release agents, can create a barrier between the metal surface and the electroplating solution. This barrier can trap gases released during electroplating, leading to the formation of blisters. Hydrogen evolution can also contribute to blistering. During electroplating, hydrogen gas is produced at the cathode (the zinc die-cast part being plated). If the electroplating process is not properly controlled, excessive hydrogen generation can occur. Hydrogen can become trapped in pores, imperfections, or microstructures on the surface of the die-cast part, leading to the formation of blisters as the gas accumulates. Inadequate cleaning can likewise impact blistering. Proper cleaning and the preparation of the zinc die-cast part before electroplating are crucial to removing any contaminants and ensuring good adhesion of the electroplated layer. Inadequate cleaning can leave behind impurities that contribute to blistering. Finally, porosity in the die-cast can affect blistering. Zinc die-casting processes can sometimes result in parts with porosity, having tiny voids or pores within the material. If these pores are not properly sealed or filled before electroplating, they can act as sites for gas entrapment, leading to blistering.

4. Conclusions

The microstructure layers of specimens B1, B2, and B3 can be categorized into three regions: chill region, columnar region, and equiaxed region. The thickness of each region depends on the cooling rate of the specimen. Additionally, the thickness of the chill area is influenced by the duration of polishing of the workpiece before plating. During examination using Backscattered Electron Imagery (BEI) and Secondary Electron Imagery (SEI), it was observed that voids formed at the discontinuity mark beneath the area where the surface blistering and pores were distributed throughout the workpiece. The microstructure consists of two types of structures and phases: the η phase, characterized by a round white appearance, and the eutectic structure, which is a mixture of two phases, namely the η phase and α phase, appearing as scattered white and black stripes. A layer of discontinuity is present in the zinc layer, located beneath the blistering surface. This suggests that the blistering of surface

from the high-pressure casting is caused by gas trapped within the discontinuity layer, when stimulated by external factors such as heat causing the expanding gas to push the material outwards. Gas chromatography was used to examine the gas content in the workpieces, revealing the presence of hydrogen gas with an average concentration calculated at 0.00755 percent/weight in each workpiece sample. Controlling gas production in casting workpieces, particularly in high-pressure casting processes, is typically challenging.

Acknowledgments

This research project is supported by Mahidol University.

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