

STUDY OF HARDNESS AT DIFFERENT LOCATIONS OF CAST AL/TiB₂ MMC CAST INGOT BASED ON MICROSTRUCTURES

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Abstract: The present study deals with aluminium-based in-situ composites wherein the reinforcement, TiB₂ were synthesized in the molten alloy via reaction of salts between K₂TiF₆ and KBF₄, and hence named as the mixed salt route. The concept of in-situ composites is an offshoot of the technique used to produce grain refiners for aluminium alloys using similar salts. The paper shall cover a few aspects of this new generation of composites, such as development of in-situ composites and effect of holding time at the pouring temperature of TiB₂ on hardness in Al/TiB₂ Metal Matrix Composite (MMC). Totally nine cast ingots of A356 cast aluminium alloy with 6% TiB₂, in-situ formed, have been fabricated by stir casting route at three different pouring temperature of 750°C, 780°C and 810°C for holding time of 30 minutes. In these ingots the distribution pattern (grain structure) and number of TiB₂ particles present at six different identical locations each, for all the nine ingots were studied by utilizing Scanning Electron Microscope (SEM) and Optical Microscope. An analysis has been made to reason out the pattern of distribution of TiB₂ particles based on pouring temperature and holding time after mixing the halide salts to form the TiB₂ in-situ inside the A356 aluminium melt. The distribution pattern of TiB₂ particles at various locations as obtained from SEM micrographs and Optical images respectively, in various locations have been inferred. The macro hardness values were measured by using Brinell hardness tester at each of the six locations of nine ingots of Al/TiB₂ MMC. The number of replications was three for all the macro hardness measurements.

Keywords: Metal Matrix Composite; Hardness; In-situ; TiB₂; MMC

1. Introduction

Aluminium based metal matrix [AlMMC] composites are preferred because of the increased tensile strength,

hardness, fatigue strength and elastic modulus or stiffness as compared to the base matrix alloy [1,2]. These AlMMCs are fabricated by adding ceramic reinforcement particles such as SiC, Al₂O₃, TiC, CBN and TiB₂. Out of these reinforcements TiB₂ is considered to be the most superior because of the maximum tensile strength, compressive strength, elastic modulus and hardness it possesses [3].

For producing Metal Matrix Composites (MMCs) by in-situ processing provide remarkable advantages over the traditional processing from both technical and commercial perspective. The skill of Salt-Metal reactions is a pretty in-situ approach evolved generally for preparing Al/TiB₂ MMCs. It includes adding mixed Ti and B bearing salts (i.e. K₂TiF₆, KBF₄) to molten aluminium, giving rise to the formation of a dispersion of TiB₂ particles in the Al matrix.

The process for evolving MMCs is based on a deep-rooted knowledge for manufacturing Al-Ti-B master alloys for the grain refining production of aluminium [4,5]. The technology practices the reaction between molten salts to produce TiB₂ in-situ within molten aluminium. The master alloy consist of a fine distribution of TiB₂ in aluminium but at a much diluted level. The products would combine high strength with good ductility and would be competitive both technically and economically with existing cast MMCs [6,8]. This really affords a novel low-cost method for manufacturing a kind of valuable composite.

The uses of this type of material are predicted in different parts of the automotive and aerospace industries. The TiB₂ particles, in-situ formed in the matrix of pure Al alloy are very fine in size. They usually stay close seeming as strings or clusters of particle agglomerates, leading to an inhomogeneous distribution [9,12]. At the present time no literature can be found reporting on cluster free TiB₂ particles. In order to achieve a beneficial composite with good properties, we have to achieve less agglomeration and preferential distribution pattern and number of TiB₂ particles. One methodology for this purpose is to discover an appropriate alloying element for the matrix

aluminium which could definitely modify the interface

The objective of this paper is twofold. The first one is to study the influences of processing parameters on the kinetics of TiB_2 creation as well as on the microstructure (grain structure) of the composite. The second is to increase the hardness value of TiB_2 MMC by means of chemical additions in the aluminium matrix.

2. Experimental Work

2.1 Materials

The two halide salt powders potassium hexa fluoro titanate and potassium tetra fluoro borate (K_2TiF_6 and

properties between TiB_2 and Al [13,16].

KBF_4) were procured. The particles of the two different salts are irregularly shaped. Commercial cast aluminium ingot bars machined into small chips were used as base metal to synthesize Al/ TiB_2 MMCs. In this work to improve the dispersion, Al binary alloys were used as the base metal for processing the in-situ composites. The examined alloying elements were Mg, Mn, Cu, Zn, Fe, Ti and Si were shown in table.1 [17,18].

Table 1. Chemical composition of A356 alloy (wt. %)

Si	Fe	Cu	Mn	Mg	Zn	Ti	Al
6.5-7.5	0.2	0.2	0.1	0.25-0.45	0.1	0.1	Balance

2.2 Preparation of the samples

The nine Al/ TiB_2 cast ingots were prepared by stir casting route. Initially pre calculated amount of chips (3 Kg) of the commercial pure aluminium (A356) were loaded in a graphite crucible and melt in a furnace atmosphere at a set temperature up to 850°C . Then the calculated amounts of halide salts potassium hexa fluoro titanate and potassium tetra fluoro borate (K_2TiF_6 and KBF_4) were slowly added to the Al melt. The stirring is continued up to next 25 minutes, and Ti and B elements

in the salts diffuse in the presence of aluminium to form TiB_2 .

An aluminium and salts were melted in a graphite cylindrical crucible under flowing Argon gas atmosphere with pouring temperature and holding time as parameters. All with combinations of the nine ingots as pouring temperature of 750°C to 810°C with holding times of 30 minutes after mixing the salts [19,20] were thus cast and the experimental set-up is shown in Fig.1.

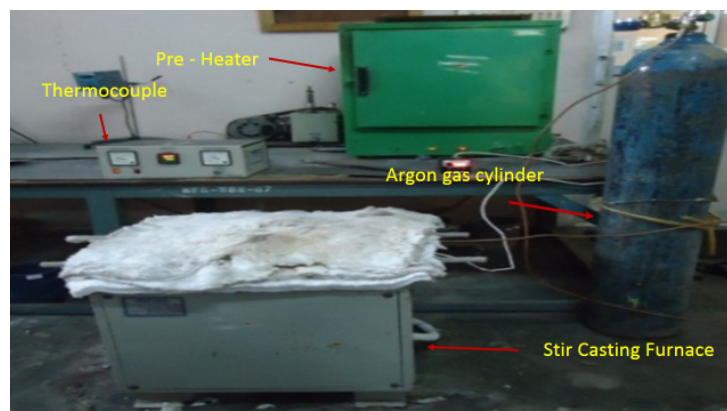


Figure 1. Schematic diagram of the experimental set-up.

2.3 Evaluation of the castings

After solidification of each casting in the permanent mold (die-mold) Specimens of $10 \times 10 \times 10$ (mm x mm x mm) were cut at six locations, as shown in Fig. 2.

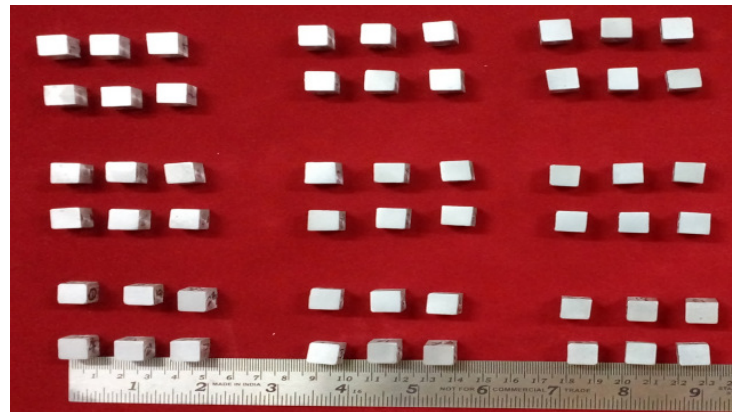


Figure 2. Al/TiB₂ MMC specimens for SEM and Hardness

After machining the samples were etched in Keller's etchant (HF 2 ml + HCl 3 ml + HNO₃ 5 ml + H₂O 190 ml). The microstructure was then

characterized by Scanning Electron Microscope (JEOL-JSM 6610LV model), as shown in Fig.3.

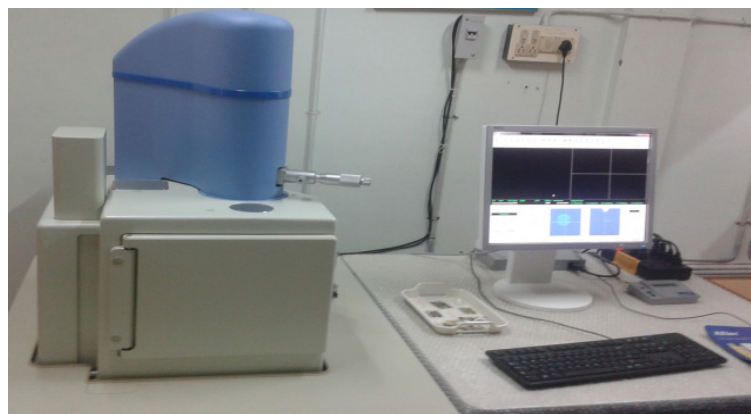


Figure 3. Scanning Electron Microscope (JEOL-JSM 6610LV model)

The transient temperature distribution affects the microstructure very much. Both SEM and optical micrographs show significant variation.

2.4 Hardness

The ingots were dimensionally marked as 24 locations from top to bottom, as shown in fig 4. Out of the 24, six locations were particularly selected in the ingot (top, middle and bottom) and the specimens were subjected

for hardness measurement by using the Brinell hardness tester with the description (load = 100 kgf, ball diameter = 1/16 inch, scale =B and supplied by M/s. Madras Metallurgical services, Chennai). The Brinell hardness tester is shown in fig 5. Three replication hardness values were found on each sample. The average hardness values were calculated from the three replication values, they were tabulated for all the pouring temperature with 30 minutes holding time, it is shown in the Table 2.

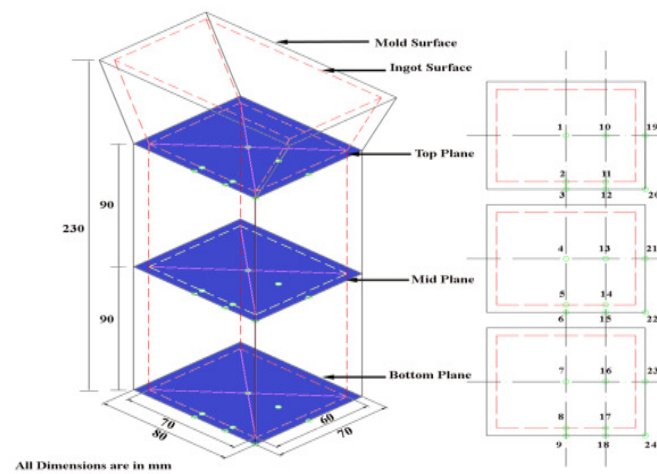


Figure 4. Dimensional drawing of ingot cast, shown with 24 different locations considered



Figure 5. The Brinell hardness tester

Table 2. The average hardness values for all the pouring temperature with 30 min holding time

Location	Pouring Temperature											
	750 °C			780 °C			810 °C			750 °C	780 °C	810 °C
	Hardness value from three replication, BHN									Average Hardness, BHN (Round off value)		
	1	2	3	1	2	3	1	2	3			
4	59	59	62	62	63	61	58	59	57	60	62	58
23	64	66	65	66	68	67	63	63	63	65	67	63

3. Results and Discussions

3.1 Formation of Al/TiB₂ products

3.1.1 Mechanism of growth in size of TiB₂ particles

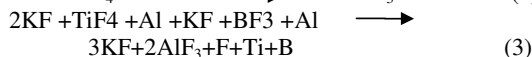
When the titanium bearing and boron bearing powder particles come in contact during mixing in aluminium melt the reaction might begin releasing fluron gas. At the same time due to the exothermic reaction occurring locally between the salt powder particles the temperature will shoot up enormously forming TiB₂ molecules.

Between the TiB₂ molecules the fluron gas formed as a byproduct will try to escape out causing low pressure between the TiB₂ molecules. The surrounding aluminium liquid will eventually consolidate the TiB₂ molecules together and a bigger TiB₂ particle will result and slowly with time the surrounding TiB₂ molecules formed fresh will attach themselves to the TiB₂ particle already formed and growth in size may thus take place. This growth in size will cease when the reaction is complete.

3.1.2 Mechanism of In-Situ Reaction

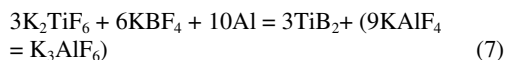
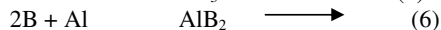
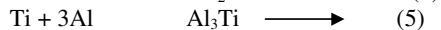
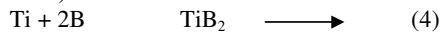
In this process, mixed salts of potassium hexa fluoro titanate (K₂TiF₆) and potassium tetra fluoro borate (KBF₄) are introduced into an aluminium melt which react with liquid aluminium, forming TiB₂ precipitates within aluminium. Exothermic reaction mechanisms were started with the stoichiometric ratio 1:2 of two types of salts. It can be seen that TiB₂ cannot be directly formed from the reduction of K₂TiF₆ and KBF₄.

First, K₂TiF₆ and KBF₄ in the Al melt are separately decomposed to KF liquid, titanium fluoride and boron fluoride gases. Then Ti and B ions are released and diffused into liquid Al through the aluminothermic reduction of titanium fluoride and boron fluoride gases at the molten flux-liquid metal interface.



Where, by products KF, F, AlF₃ are not useful and were removed as slag or natural volatilization (a substance that is easily evaporated at normal temperature) prior to casting.

In the second step of the reaction, the solutes Ti and B in liquid Al reach saturation, they will be separated out as the intermetallic compounds TiB₂, Al₃Ti and AlB₂ according to equations (k Niranjana 2015).



The TiB₂ particle formed may not be completely in solidified form before pouring and the TiB₂ particles formed may be in the process of solidifying, and thus, may be in semi-solid condition during pouring.

When the mold is poured from a particular height the turbulence is expected to be higher locally at the bottom most points of the ingot and this may cause the semi-solid (freezing) TiB₂ particles getting fragmented and the TiB₂ particles found in these surroundings may be smaller in size.

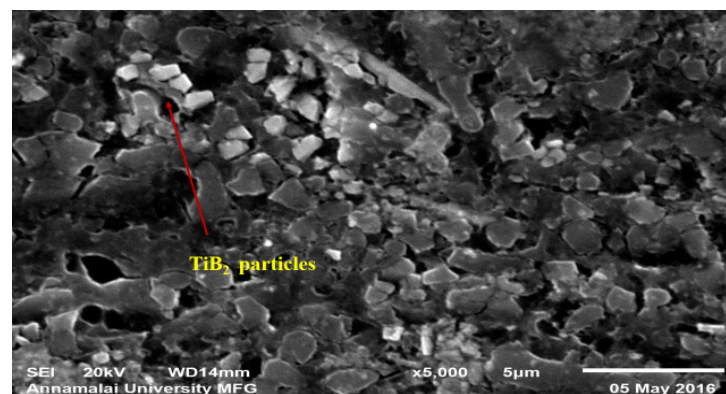
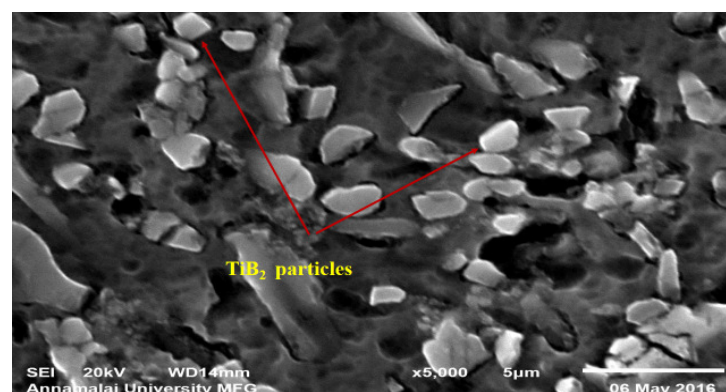
Whereas, at the top of the ingot during filling the height of fall of liquid MMC from the ladle to the mold will be less and hence turbulence also will be minimum at the locations near the top of the mold and hence the TiB₂ particles may not get that easily fragmented and the size of the TiB₂ particles observed in these locations may be bigger [22].

Likewise, location – 23 is at the bottom near the mold surface of the ingot. At this point the fluidity is maximum at 810 °C and the cooling rate is also found to be the maximum. Because of this and as the fall through height of the liquid metal while filling is maximum for location – 23 the turbulence will be maximum and because of this the TiB₂ particles are smaller and maximum at this point, as shown in table 4.

The pouring temperature 810 °C hardly any variations in the microstructure are observed as found in table.3. The percentage relative area of reinforcements is listed here. The XRD images confirm the presence of TiB₂ as shown in Fig.7.4.

Table 3. Percentage TiB_2 particles in six locations of ingot from SEM micrograph

Location as per figure - 1	Temperature in $^{\circ}\text{C}$	Particle size for holding time in μm
		30 minute
1	750	35 - 40
	780	35 - 40
	810	40 - 50
4	750	35 - 40
	780	30 - 40
	810	40 - 50
7	750	25 - 35
	780	40 - 50
	810	60 - 70
19	750	30 - 40
	780	40 - 50
	810	50 - 60
21	750	40 - 50
	780	30 - 40
	810	60 - 70
23	750	40 - 50
	780	50 - 60
	810	70 - 80

**Figure 6.1.** SEM image for pouring temperature 810°C at location 1**Figure 6.2.** SEM image for pouring temperature 780°C at location 4

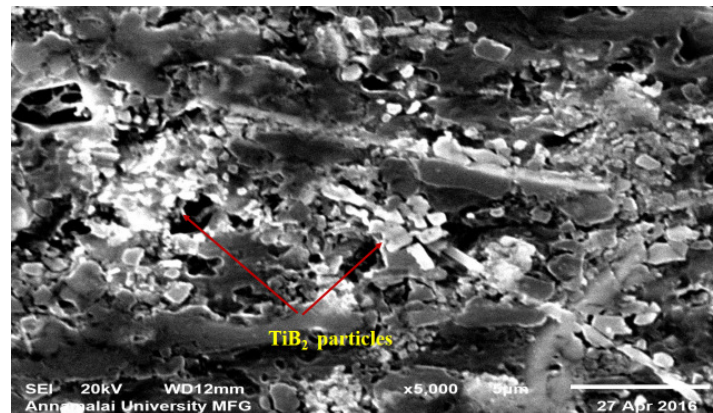


Figure 6.3. SEM image for pouring temperature 810 °C at location 23

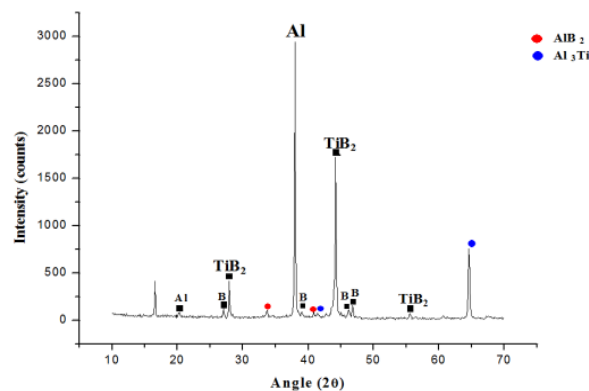


Figure 6.4. The XRD images for Al/TiB₂ MMC

3.1.3 Influences of holding time

The holding time 30 minutes cause some variations in the size of TiB₂ reinforcement particles. The SEM

images from fig. 6.1 to 6.3 shows the particles size developed and their crystal boundaries were formed better when increasing the pouring temperature, as found in table. 4.

Table 4. TiB₂ particle size in microns at six different locations

Location as per figure - 1	Temperature in °C	Particle size for holding time in µm
		30 minute
1	750	1 – 1.5
	780	1 – 1.5
	810	1.5 - 2
4	750	1 – 1.5
		L – 5, D -1
	780	1 – 1.5
7	810	1 – 1.5
	750	1 – 1.5
	780	1.5 – 2

	810	1 – 1.5 L – 8, D -1.5
	750	1 – 1.5
	780	1 – 1.5
19	810	1 – 1.5
	750	1 – 1.5
	780	1 – 1.5
21	810	1 – 1.5
		L – 10, D -1.5
	750	1.5 – 2
	780	1 – 1.5
23	810	1 – 1.5

Where,

L = Length of needle shaped TiB_2

D = Diameter of needle shaped TiB_2

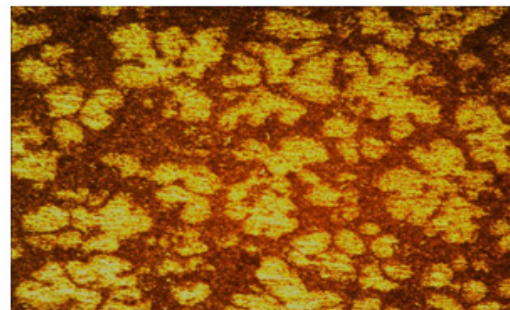
3.2 Influences of hardness

3.2.1 Distribution of TiB_2 particles

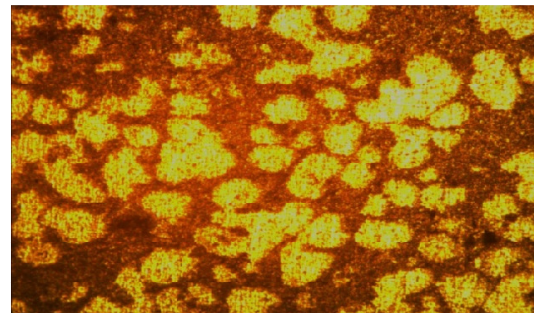
While filling, near the surfaces of the mold the heat transfer rates will be very high and this may cause lower temperature of the liquid metal near these surfaces and at the center points of the ingot the heat transfer will be lower and temperature of the liquid metal will be higher at these locations.

Due to the variation in temperature between the center and surfaces a circulation of liquid metal may be set up due to density differences. Near the surfaces the liquid metal will cool drastically and may be mushy near the surfaces and on further circulations more TiB_2 particles will be trapped in the mushy liquid near the surfaces.

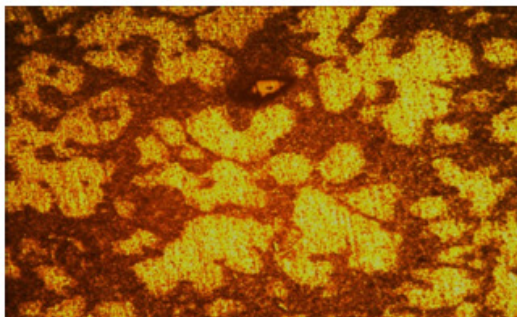
This may be reason for the presence of more number of fine TiB_2 particles near the surfaces than near the center points. Hence at near the surfaces of the ingot have more number of TiB_2 particles available and it is small and fine structured (fig 7).



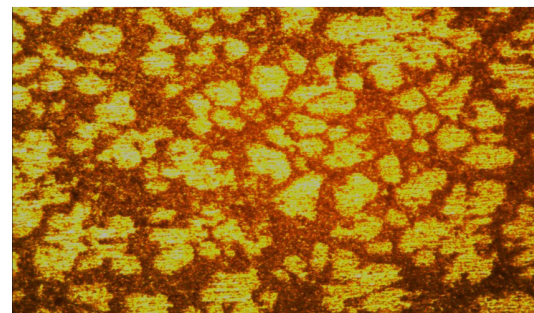
For 750 °C with 30 minutes at location 23



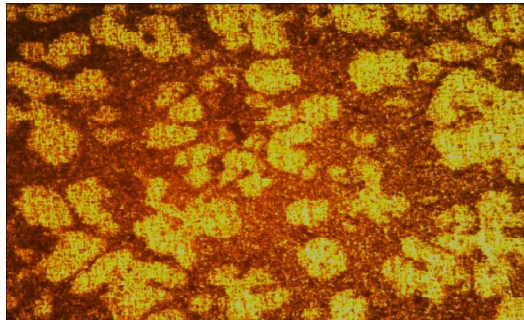
For 780 °C with 30 minutes at location 4



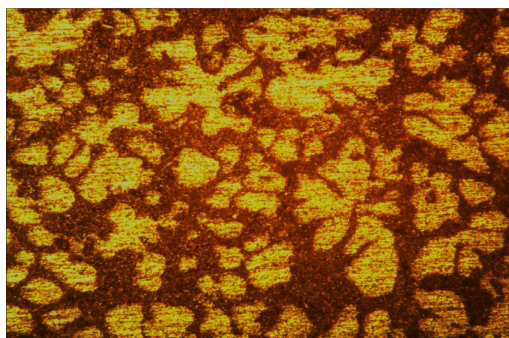
For 750 °C with 30 minutes at location 4



For 780 °C with 30 minutes at location 23



For 810 °C with 30 minutes at location 4



For 810 °C with 30 minutes at location 23

Figure 7. Optical microscope image for pouring temperatures from 750 °C to 810 °C at locations 4 and 23 of the ingot.

Therefore at this location the hardness values are higher than the center point location of the ingot. The hardness values for the different locations of the ingot were tabulated in (Table 2).

4. Conclusions

At location -23 as the TiB_2 particles are smaller due to the fact that while pouring, as the fall through height for the liquid MMC is more, the turbulence would have been maximum and the bigger TiB_2 particles would have got fragmented. As the TiB_2 particles are found to be finer they will impede dislocation movement and hence the local strength and hardness will be more.

At location - 4 as the fall through height for liquid MMC is less the TiB_2 particles are bigger at this location, as found in the optical micrographs. Bigger the TiB_2 particles, they will not impede the dislocation movement and hence the hardness is less at this location.

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