

# Characterization and Investigation of Grain Selection in Spiral Grain Selectors during Casting Single-Crystal Turbine Blades

Hassan Gheisari<sup>\*1</sup>, Ebrahim Karamian<sup>2</sup>

<sup>\*1</sup>Department of Mechanical and Material, Lenjan Branch, Islamic Azad University, Isfahan, Iran

<sup>\*</sup>Email of Corresponding Author: Gheisary@iauln.ac.ir and Tel: +989135344540

<sup>2</sup>Advanced Materials Research Center, Faculty of Materials Engineering, Islamic Azad University, Najafabad Branch, IAUN, P.O. Box 517, Isfahan, Iran

*Received: June 12, 2015, Accepted: September 5, 2015*

## Abstract

Manufactured single crystal components using Ni-base super alloys are routinely used in the hot sections of aero engines and industrial gas turbines due to their outstanding high temperature strength, toughness and resistance to degradation in corrosive and oxidative environments. To control the quality of the single crystal turbine blades, particular attention has been paid to grain selection, which is used to obtain the single crystal morphology from a plethora of columnar grains. For this purpose, different designs of grain selectors are employed and the most common type is the spiral grain selector. A typical spiral grain selector includes a starter block and a spiral (helix) located above. It has been found that the grains with orientation well aligned to the thermal gradient survive in the starter block by competitive grain growth while the selection of the single crystal grain occurs in the spiral part. In the present study, 2D spiral selectors with different geometries were designed and produced using a state-of-the-art Bridgman Directional Solidification casting furnace to investigate the competitive growth during grain selection in 2d grain selectors. The principal advantage of using a 2-D selector is to facilitate the wax injection process in investment casting by enabling significant degree of automation. The directional solidification process for obtaining single crystal component for Nickel Super alloys based on the competitive growth in 2D and 3D single crystal grain selector was experimentally investigated for various geometries. Transverse sections of the grain selectors using optical and EBSD microscopy techniques were observed to understand the competitive grain growth process.

## Keywords

Grain Selector, Single Crystal, Directional Solidification, CMSX-4 Super alloys, Investment Casting

## 1. Introduction

Single crystal turbine blades are produced using Nickel base Super alloys consisting of complex multi-phase microstructure. These blades are commonly used in hottest sections of industrial gas turbine and jet engines for their outstanding high temperature in-service strength, creep and thermal fatigue resistance and ability to resist degradation in severe high temperature conditions. The increasing demand for turbine inlet temperatures over the past three decades meant that manufacturers face difficult task of continuous quality improvements of Nickel based turbine blades. Material design and process control play a vital role in developing heat resistant materials that possess a greater tendency to perform in elevated temperatures. Increasing the operating temperature in jet engines is desirable as it results in greater energy savings and higher efficiency [1]. This analogy has given impetus to manufacturers to develop Super alloys with optimized

thermal-mechanical properties.

Directional solidification (DS) casting techniques based on Bridgman crystal growth are used to cast the turbine blades due to material being impossible/ difficult to forge or weld. One of the desirable aspects of single crystalline components is the mechanical strength at higher operating temperatures, and is significantly superior compared to that of polycrystalline components [2]. SX components have excellent strength along the length of the turbine blade, whereas the creep strength need not be isotropic since the stress will majorly be unidirectional during its operation. This will also result in exceptional resistance to fatigue failure. SX also eliminates the grain boundaries previously found in equated and columnar castings, responsible for limitation for creep ductility [3, 4].

As a result, single crystal turbine blades have replaced the poly crystalline turbine blades entirely on the basis of possessing greater mechanical strength at higher temperatures [2, 5]. Single crystal casting is generally produced by either seeding method or grain selector method. Seed with desired crystal orientation is placed at the bottom of the mold in the seeding method [6]. The second method is the grain selector method in which, a grain selector is placed on a chill plate for nucleation of randomly oriented grains. During the grain growth in a grain selector, automatic grain selection continues to take place until one grain survives that goes on to be the single crystal (SX) with the preferred growth direction to be nearest to  $\langle 001 \rangle$ . The process of grain selection using a grain selector is of random nature since the surviving grain is not always optimal. The objective here is to consistently produce turbine blades with single crystal aligned closer to the  $\langle 001 \rangle$  direction along the length of the blades [7-10]. However there remains some inconsistency during the process, as it does not always yield such blades with desired orientation.

Spiral grain selectors are majorly used in industry to manufacture turbine blades for gas and aero engines out of single crystal Super alloys; therefore it is critical to control its grain orientation. A helical pattern with cylindrical base is employed to control the grain orientations in blades during DS process. Previous studies [11, 12] have shown that the role of spiral part is to efficiently select a well-orientated grain by a mechanism called “geometry blocking” while the primary grain orientation optimization takes place in the starter block.

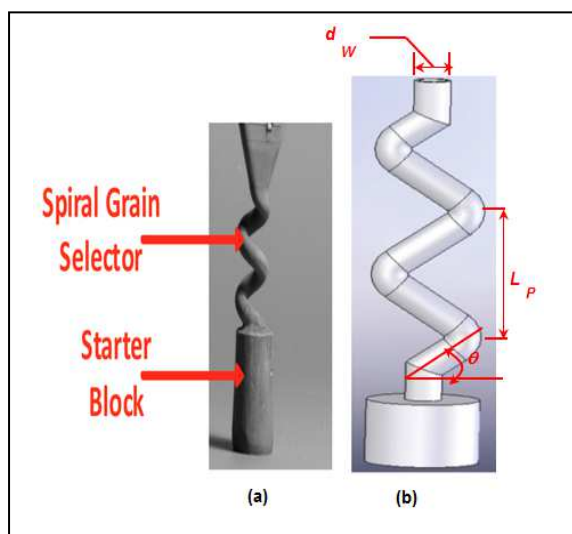


Figure 1(a). Illustration image of single crystal grain selector [13] (b). 2D grain selector with parameters

Figure 1(a) shows a conventional grain selector, the upper section is the spiral selector and a cylindrical base is the starter block. Recently Dai [12-14] investigated the role of 3 dimensional spiral parts on grain selection and concluded that the geometry of the spiral grain selector has greater influence on the efficiency of the grain selection process. The efficiency of spiral decreased considerably by increasing the take-off angle and therefore concluded that the take-off angle should be preferred in the region of 25 and 30 degrees. Smaller wax wire diameter and take-off angle (various parameters showing in Figure 1(b)) reduces the height of SX selection, while the spiral does not play any role in optimizing the grain orientation but randomly selects a single grain. Esaka [15] developed a two dimensional analytical model depending upon the theory of columnar dendrite growth. The grain selection process efficiency was studied for the 2-D model in terms of height of starter block, initial number of seeds and “pig-tail” width, take off angle and length which greatly influences the present work since any experimental work has not been conducted due to time and cost constraints. The aim of this study is to conduct a systematic study on the grain selection process in 2-Dimensional grain selectors to improve the understanding and hence the efficiency of the process. For this study, series of casting trials were performed with newly designed 2-Dimensional grain selectors based on various take off angles, wax wire diameter and zigzag width to understand the single crystal texture evolution occurring in the zigzag section. The dimensions were kept as nearest to the parameters currently used in industry to maintain the consistency of the process.

## **2. Experimental methods**

### *2.1 Review Stage*

Investment casting trials were carried out using a Bridgeman type DS induction Furnace located at University of Birmingham, UK (IRC). Various 2D Perspex grain selector based on CAD design are using a process called rapid prototyping. This process produces 3D model/designs from a CAD file to build accurate prototype part/models for verification of a product design. A computer-controlled laser is used to cure the liquid resin (Somos 8110) that is then stacked up from the base layer by layer to produce the part.

A wax model is generally prepared by injecting molten wax using a Mueller Phipps wax injection machine into a prepared master metallic mold dye. Cylindrical bars and base of the assembly were produced using the mold dye. For this project, the produced 2D Perspex grain selectors produced using rapid prototyping were dipped in Blaysons A7/FR60 pattern molten wax for approximately 4 seconds and left to dry, to build a layer of wax. The wax surface was then smoothed to remove any visible blobs of wax. The wax spirals were then placed on a circular carousel. Four spirals as shown in Figure 2(a) were arranged in circular configuration consisting of feed arrangements and test bars. The test bars were cylindrical (of dimension 15mm dia x 600mm length) and were attached to 2D grain selector. The cylindrical downpour was placed on top of the assembly to maintain firm structure hold with test bars during ceramic coating process. The regions of intersection of wax parts were smoothly polished to eliminate any cracks or holes on the surface.

The following stage is called investment shelling. The wax assemblies were dipped into ceramic slurry using Motoman Ceramic Shell manufacturing machine. This gave a uniform ceramic coating across the surface. The ceramic slurry consists of binding agents and mixture of Zircon Silicate and

Silica sol. The wax assembly is then stuccoes with larger ceramic particles of Zircon filler. The assembly was allowed to dry for 3 hours to allow the binding of particles properly to the surface. This operation was repeated four times to form a thick strong layer ceramic shell to ensure that it is not susceptible of cracking. The full ceramic shell assembly is shown in Figure 2(a).

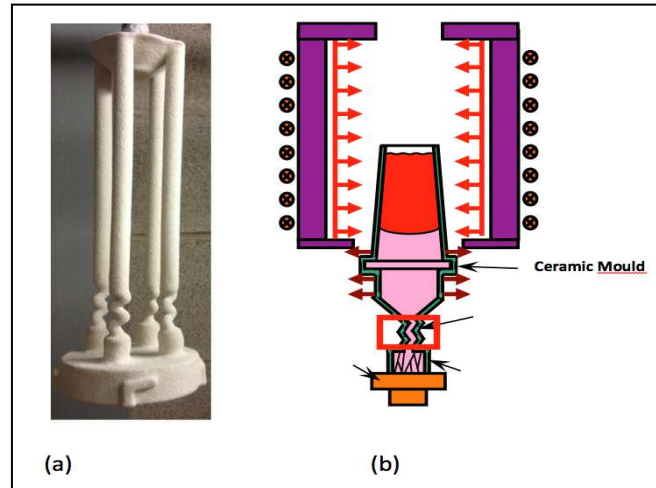


Figure 2(a). Assembly mold with ceramic coating; (b). Schematics of Investment Casting Furnace for Single Crystal Castings [13]

Once the shells were fully prepared, the next stage was to de-wax the molds. The ceramic shells were placed in a LBBC Boilerclave unit for 45 minutes at around 600 C to de-wax the mold. The assemblies were then fired at 800 C for 60 minutes in autoclave to burn out the SLA constituting the spirals and partially sinter the other part of the shell. Once the shell assemblies were cooled-off, a colored dye was filled in the hollow shell to inspect any cracks in the mold. The mold was now ready for the casting process.

An industrial directional solidification furnace (Figure 2(b)) at University of Birmingham (U.K) was used to cast the samples. Prior to casting, the furnace chamber was evacuated to a partial pressure of 0.001 Pa and the mold was raised into the furnace chamber, which was preheated to 1500 C using graphite resistance elements. The furnace was operated in vacuum condition to prevent any reaction between the molten alloy and furnace atmosphere. The mold was mounted on the water-cooled chill plate to remain in direct contact to the base to initiate grain nucleation. It was held for 20 minutes to soak [4]. The charge (CMSX4) was induction melted and was poured in the hollow mold. The ram on which the mold was placed then gradually lowered at the withdrawal rate of 3.81 mm/min. Low pulling velocity and high positive thermal gradient are desirable to promote competitive growth. Planar growth front was maintained using water-cooled baffles that subsequently maintain the heat flux perpendicular to the direction of withdrawal. In total, three molds were produced each taking approximately three hours to cast entire assembly.

The alloy used in this study was nickel based super alloy CMSX4 with nominal composition of 3.0 Re, 6.5 Cr, 9.5 Co, 0.6 Mo, 6.0 W, 6.5 Ta, 5.6 Al, 1.0 Ti, 0.5 Hf and balance Ni (Wt%) [11]. To analyze the grain microstructure within the grain selector, the samples were cut at various points of interests at certain heights. The samples were polished to achieve 1 micron m surface finish with final polishing stage using colloidal silica and water, each for 20 minutes to obtain deformation free surface for EBSD analysis. FEI Sirion 200 FEGSEM was used at accelerating voltage of 20 kV

with spot size of 5 to observe the grain structure and to gather orientation evolution maps. These were later post processed using HKL Channel 5 (Oxford Instruments) software to obtain further data such as Inverse Pole Figures (IPFs) and automatic grain mapping and indexing.

### 3. Results and Discussion

#### 3.1 Grain Selection in Spiral/2D Zigzag Grain Selector

The zigzag single crystal grain selector consists of a cylindrical starter block and a zigzag section of approximately 2 pitch length as shown in Figure 3(a). Figure 3 (b-e (top)) depicts the evolution of grain structure in spiral grain selector for sample 2. The spiral was macro etched to reveal the external surface grain structure. By visual observation, a large number of equiaxed grains are nucleated at the base of the starter block. There seemed to be a large decrease in the number of grains as the distance from the base of copper chill plate increases, simultaneously the size of the surviving grain increases. The primary reason for increase in grain size and decrease in number of grains is the progressive evolution of equiaxed grains in to parallel columnar grains along with occurrence of competitive grain growth in starter block [3]. The mis-oriented grains are blocked off by the preferred columnar grains while minimizing the deviation angle between the  $\langle 001 \rangle$  direction of grain and the direction of casting axis [16]. It was found that at the top of the starter block most grains were found to have about 8-25 degree deviation.

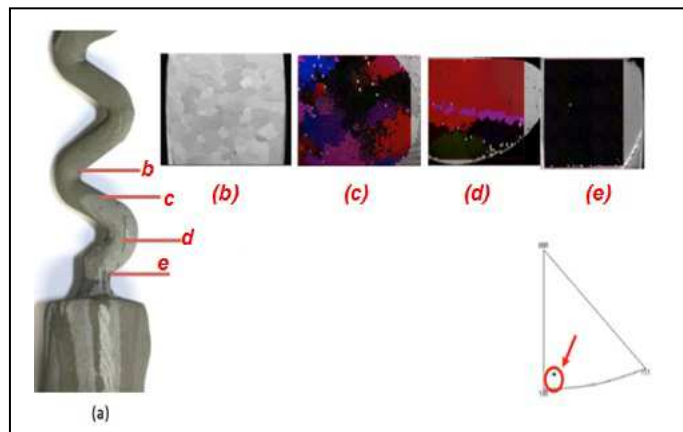


Figure 3(a). Casted 2D-Spiral selector (sample 2); (b-e). EBSD maps of the transverse sections at heights shown in (a) and IPF for final selected grain

The axial crystallographic grain texture evolution and Inverse Pole Figures (IPFs) of cross sections at final grain selected height in the zigzag part of selector are shown in Figure 3 (e (bottom)). The IPFs of  $\langle 001 \rangle$  crystallographic plane describes the deviation of grains with respect to the casting axis (vertical). At the point of intersection of the starter block and the zigzag section, a large proportion of grains disappeared when entering the zigzag passage and only a small number of grains persisted. At the height of 1 pitch length, the grain size increased leaving the number of surviving grains in the region of 10-15. The efficiency of the process can be predicted by the swiftness at which the reduction in number of grains takes place and effectiveness of the single grain selection. The examination of corresponding crystallographic texture maps clearly revealed that the optimization of grain orientation does not occur in the zigzag part and hence can offer the

chance for a mis-oriented grain to overgrow favorable oriented grains. This mainly occurs due to lack of space for expansion of dendrites and therefore grains are geometrically restricted by wall and/or turnings. For this particular sample, the grain selection occurred at about 1 pitch length with deviation of about 13 degree from  $\langle 001 \rangle$  direction. Consequently, a completely opposite phenomenon is observed in starter block, which consists a non-varying geometry that allows the competitive grain growth by allowing well aligned grains to overgrow their counterparts pertaining closer to  $\langle 001 \rangle$  axial texture. This concludes that the role of zigzag is to reduce the number of grains, but successfully select single crystal with no tendency to optimize grain orientations [11].

### *3.2 Geometry of ZigZag Selector*

The grain selector geometry in zigzag section such as the take-off angle and wax diameter impose a significant influence on the grain selection process during investment casting. It is known that larger take off angles will result in decrease of grain selection efficiency [10, 11] therefore take-off angles of 30 and 40 were used in the first casting trial. As expected, the grain selectors with 40 degrees take off angles resulted in polycrystalline samples during the experimental casting trials. Dai et al. [11] concluded that by increasing the take-off angle, thermal flow conditions will dominate the grain selection process, while entirely nullifying the effect of geometry restriction (wall) is making it inefficient. A thicker wax diameter creates manufacturing difficulties to prepare wax zigzag selector mold for smaller take off angles of 25 degrees or less; therefore it is desired to select take-off angles in the range between 25 and 30 degrees (inclusive) in industrial SX casting process. The height of the single crystal selection increases with increase in the wax wire diameter. As the wire diameter decreases, the space decreases which allows fewer grains to enter the zigzag section and restricts dendrites to branch due to lack of availability of space making grain selection process severe. The smaller wax diameter (less than 3 mm) poses rigidity risk on the assembly and therefore loses the strength to support the weight of full wax assembly, hence, arising chances of failure during ceramic coating process whereas a thicker wax diameter (>5mm) has practical manufacturing limitations on zigzag turnings at lower take off angles.

### *3.3 Modes of Grain Selection*

The competitive grain growth theory in directional solidification somewhat does not apply especially in the spiral/zigzag part where the direction of the solidification front suddenly changes at the turnings. The axial texture in this case is also absent due to range of axial orientation often deviating in region between 12-20 degrees from  $\langle 001 \rangle$  direction, hence a better understanding is necessary for the process of grain selection in spiral/zigzag. From earlier discussion it is imminent that, geometry plays a vital role in the final selected single crystal process and hence it can be concluded that geometrical blocking mechanism dominates [11, 14]. A typical example that represents the geometrical blocking mechanism in the grain selector is presented in Figure 4 depicting various geometries of grain selector.

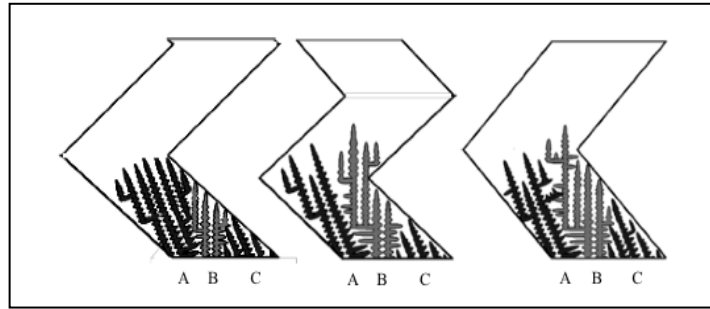


Figure4. Examples (1, 2, 3) of geometrical blocking mechanism in grain selectors [13]

The characters A, B and C in Figure 4 represent grains with different orientations, respectively. Grain B is favorably oriented with the casting axis closer to heat flow direction, whereas the primary dendrites of grains A and C are slightly off-axis and make an angle of approximately 20 degrees with vertical axis. At the onset of solidification process, grain B being favorable aligned grows ahead of the other two competing grains. This allows grain B to expand by branching secondary and tertiary dendrites from the primary dendrites due to availability of free space. In the case of example 2 and 3, grain A being behind is finally blocked by the zigzag wall on left, whereas grain C being mis-oriented is overgrown by grain B. The primary dendrite of grain C impinges on the right hand side of not only mold wall but also dendrite trunks of grain B. The primary dendrite of grain C not only impinges on the right hand side of mold wall but also dendrite trunks of grain B. Whereas in example 1, grain B being well aligned is blocked by inclined mold wall, undesirably allowing misaligned grain A to grow further. Reduction in pitch length (ex 2) increase turns in the selector and consequently offer more chance for geometrical blocking mechanism to dominate. Reducing the wax wire diameter (ex 1) allows less number of grains to enter the grain selector and also results in reduction of available space for grains to develop. Increasing take-off angle (ex 3) allows the grains A and B to grow in positive thermal gradient over a longer solidification height until they reach the spiral wall. Both the geometrical blocking mechanism and thermal control mechanism are responsible for final selected grain. However, for low take-off angle, the geometrical blocking mechanism dominates the grain selection process due to lack of space available for other grains to compete [13].

The spatial location of each grain within the initial grain passage of grain selector dictates the grain selection process [17, 18]. Grains entered from the starter block were blocked-off by the grains which are located close to the first lateral grain passage. It can be prematurely concluded that the grain near the inner wall of the passage is usually selected as grain i.e, the grain which follows the shortest path. The grains present near the outer wall/far away from inner wall are geometrically blocked by wall of the zigzag passage. The effective grain selection in the zigzag section is considered to be mainly due to the coupling effect of the heat flow direction and the geometrical restriction of the spiral wall [18].

#### 4. Conclusion

The directional solidification process for obtaining single crystal component for Nickel Super alloys based on the competitive growth in 2D and 3D single crystal grain selector was experimentally investigated for various geometries. Transverse sections of the grain selectors using optical and EBSD microscopy techniques were observed to understand the competitive grain growth process.

The following conclusion can be made

1. The competitive grain growth is observed in the starter block along with grain orientation optimization to acquire orientation close to  $\langle 001 \rangle$  axial direction.
2. The role of the zigzag selector is to successfully select single crystal without optimizing the axial orientation of grains. Take-off angle in the range of 25-30 degrees is desirable to make the process efficient.
3. Geometrical Blocking mechanism is the governing reason for single crystal grain selection process in grain selectors.

## 5. References

- [1] McLean, M. 1988. Directionally solidified materials for high temperature service, the metal Soc., London.
- [2] Pollock, T. M. and Tin, S. 2006. Nickel-based super alloys for advanced turbine engines: Chemistry, microstructure, and properties, *Journal of Propulsion and Power*, 22(2), 361-374.
- [3] Murakumo, T., Kobayashi, T. and Koizumi, Y. 2004. Creep behaviour of Ni-base single-crystal superalloys with various  $\gamma'$  volume fraction,"*Acta Materialia*, 52(12), 3737-3744.
- [4] Wilson, B. C., Cutler, E. R. and Fuchs, G. E. 2008. Effect of solidification parameters on the microstructures and properties of CMSX-10, *Materials Science and Engineering: A*, 479(1-2), 356-364.
- [5] Reed, R. C. 2006. *The Super alloys: Fundamentals and Applications*, Cambridge University Press.
- [6] Epishin, A. I. and Nolze, G. 2006. Investigation of the competitive grain growth during solidification of single crystals of nickel-based super alloys, *Crystallography Reports*, 51(4), 710-714.
- [7] D'Souza, N., Ardakani M. G., Mclean, M. And Shollock, B. A. 2000. Directional and single-crystal solidification of Ni-base super alloys: Part I. The role of curved isotherms on grain selection, *Metallurgical and Materials Transactions A*, 31(11), 2877-2886.
- [8] Dai, H. J., Gebelin, J. C., Newell, M., Reed, R. C., D'Souza, N., brown, P.D. and Dong, H. B. 2008. Grain selection during solidification in spiral grain selector, In *Superalloys 2008*; Reed, R. C., et al., Eds.; TMS: Champion, PA, 367-374.
- [9] Dong, H. 2007. Analysis of grain selection during directional solidification of gas turbine blades, *Proc world congress on engineering, World Congress on Engineering*, 1257-1262.
- [10] Seo, S. M., Kim, I.S., Lee, J.H., jo, C.Y., Miyhira, H. and Ogi, K. 2009. Grain structure and texture evolutions during single crystal casting of the Ni-base superalloy CMSX-4, in *Metals and Materials International.*, *Met. Mater. Int.*, 15(3), 391-398.
- [11] Dai, H. J., D'Souza, N. and Dong, H. B. 2011. Grain Selection in Spiral Selectors During Investment Casting of Single-Crystal Turbine Blades: Part I. Experimental Investigation, *Metallurgical and Materials Transactions A*, 42(11), 3430-3438.
- [12] Gao, S.F., liu, l., Wang, X. B., Zhao, J. and Fu, H. S. 2012. Grain Selection During Casting Ni-Base, Single-Crystal Superalloys with Spiral Grain Selector, *Metallurgical and Materials Transactions A*, 43(10), 3767-3775.
- [13] Dai, H., 2009. A Study of Solidification Structure Evolution during Investment Casting of Ni-based Superalloy for Aero-Engine Turbine Blades, University of Leicester.
- [14] Dai, H. J., Dai, H. J., Dong, H. B., D'Souza, N., Gebelin, J. C. and Reed, R. C. 2011. Grain Selection in Spiral Selectors During Investment Casting of Single-Crystal Components: Part II. Numerical Modeling, *Metallurgical and Materials Transactions A*, 42(11), 3439-3446.



- [15] Esaka, H., Shinozuka, K. and Tamura, M. 2005. Analysis of single crystal casting process taking into account the shape of pigtail, *Materials Science and Engineering: A*, 413-414, 151-155.
- [16] Carter, P., et al., Cox, D. C., Gandin, C. A . and Reed, R.C. 2000. Process modelling of grain selection during the solidification of single crystal superalloy castings”,*Materials Science and Engineering: A*, 280(2), 233-246.
- [17] Yang, C., Liu, L., Zhao, X., Wang, N., Zhang, J. and Fu, H. 2013. Competitive grain growth mechanism in three dimensions during directional solidification of a nickel-based super alloy, *Journal of Alloys and Compounds*, 578(0),577-584.
- [18] Wang, N., Liu, L., Gao, S., Zhao, X., Huang, V., Zhang, J. and Fu, H. 2014. Simulation of grain selection during single crystal casting of a Ni-base superalloy, *Journal of Alloys and Compounds*, 586(0), 220-229.

