

Optimisation of Sintering Parameters on densification of 420 steel parts produced by Metal Injection Moulding

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ABSTRACT: This paper presents the results of experimental investigations carried out to optimize the sintering parameters, such as sintering temperature, sintering time and heating rate on final densification of AISI 420 martensitic stainless steel components produced by metal injection molding (MIM) process. Taguchi method has been used for optimizing the process parameters for sintering of martensitic stainless steel (AISI 420) components. The various parameters were assigned to an L9 orthogonal array. It was found that sintering temperature has significant role on the final density of the sintered product. An optimum final density of 97 % of wrought material with a heating rate of 50C/min and isothermal heating at 1380 0C for 45 minutes in N2 atmosphere was achieved. Experiments were repeated for confirmation of results which are within the 95 % confidence interval.

KEYWORDS: Metal Injection Moulding (MIM), Sintering Parameters, Taguchi Method.

I. INTRODUCTION

A relatively new process in manufacturing, MIM is a fast-growing manufacturing process which combines the flexibility of the injection molding process with the strong structural integrity of sintered metal powder. It manufactures metal parts which are stronger, denser and more capable in production of complex geometry small components than other conventional manufacturing processes [1][2]. Table 1 shows the comparison of MIM process route with other net shape processes like Casting, Forming, Machining. From Table 1 it can be inferred that the density and surface finish of components with complex geometric profile manufactured by MIM Process are almost equivalent or superior to the other conventional manufacturing processes and due to close dimensional tolerance secondary machining is not required in MIM Process route.

Characteristics	MIM	Pressing	Casting	Machining
Density	90-100%	95-100%	95-99%	100%
Surface Finish	0.4 to 0.8	< 2	3	0.4 to 2
Secondary Machining	Not required	Required	Required	-
Wall Thickness	10	> 2 mm	> 5 mm	> 2 mm
Dimensional Tolerance	Close	Poor	Poor	Poor
Shape Complexity	High	Medium	Medium	High
Production Rate	High	High	Low	Low

Table 1. Comparison of MIM with other Net Shaping processes

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Today, MIM is serving critical performance applications in a wide range of industries and products including, automotive, aerospace and defense, cellular telephones, dental instruments, electronic heat sinks and hermetic packages, electrical connector hardware, industrial tools, fiber optic connectors, fluid spray systems, hard disk drives, pharmaceutical devices, power hand-tools, surgical instruments, and sporting equipment. connectors, fluid spray systems, hard disk drives, pharmaceutical devices, power hand-tools, surgical instruments, and sporting equipment. The higher densities of injection molded parts give much higher strength and other superior mechanical properties. The alloy AISI 420 stainless steel is one of the most widely used materials for manufacturing of knives, surgical instruments, fasteners, shaft sleeves, spindles, nozzles, shafts etc. Therefore optimization of parameters of MIM process to get high density is an important step to achieve desired mechanical properties for any specific application of AISI 420 Stainless steel. Among the three MIM processing steps, sintering is the most important step that affects the final density as well as the mechanical properties. Literature reveals ⁷⁻⁹ that the important factors of the sintering cycle are: rate of heating, sintering temperature, sintering time and sintering atmosphere. To optimize the sintering process, it is thus essential to have the knowledge on the effects of the above factors on the final density and mechanical properties. Taguchi's method is a well established statistical technique being used for design of experiment for optimization of process parameters. Fewer experiments are required in this technique than the classical design of experiments and which will enable to draw conclusion with a statistical level of confidence.

This paper presents the result of a study of the sintering characteristics of 420 stainless steel parts using the Taguchi method. The effects of three process parameters of sintering namely: rate of heating, sintering time and sintering temperature on the final density were investigated. The optimum sintering condition was proposed and confirmation experiments were conducted.

II. MATERIAL AND METHOD

MIM works with an expanding array of metal alloys. Traditional MIM parts compete well in niche markets where parts weigh less than 200 gm and wall thicknesses range from 0.25 to 6.25 mm. Tolerances are typically held to +/-0.3% and surface finishes of 0.8 mm are common. MIM parts can also be brazed, soldered, and welded as well as plated or ground to size if necessary.

Almost any metal that can be produced in a suitable powder form can be processed by MIM. Aluminium and magnesium are exceptions because the adherent oxide film that is always present on the surface of powder particles inhibits sintering. The list of metals that have been used in metal injection moulding includes many common and several less common metals and their alloys - plain and low alloy steels, stainless steels, high speed steels, copper base alloys, nickel and cobalt base superalloys, titanium, inter metallic's, magnetic alloys, refractory metals and hard metals (cemented carbides).

In this paper AISI 420 martensitic stainless steel material has been used to manufacture components by MIM process to study the densification and optimisation of sintering parameters. Martensitic stainless steels are essentially alloys of chromium and carbon that possess a martensitic crystal structure in the hardened condition. They are ferromagnetic, hardenable by heat treatments, and are usually less resistant to corrosion than some other grades of stainless steel. Chromium content usually does not exceed 18%, while carbon content may exceed 1.0 %. The chromium and carbon contents are adjusted to ensure a martensitic structure after hardening. Excess carbides may be present to enhance wear resistance or as in the case of knife blades, to maintain cutting edges.

The Composition of a typical AISI 420 grade martensitic stainless steel are:

C -0.15 %min. Mn -1% max. Si -1% max. P- 0.040% max. S- 0.03% max. Cr- 12.00% to 14.00%

The AISI 420 Martensite stainless steel powder used in this experiment is produced from Gas Atomisation process where the grains are spherical in shape and are about 5 -20µm in size.

The processing steps in MIM are:

- (1) Preparation of feedstock: Polymers and lubricant are mixed to the fine metal powder to produce a "feedstock" which is the initial raw material of MIM process. Feedstock is then converted into small pellets for easy feed in the injection molding machine.

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(2) Molding: The pellets are fed through a hopper into the screw conveyor of injection molding machine where they are converted into viscous mass with the help of heat and shear action of screws. The viscous mass is then injected inside mould cavity to produce complex geometry green compact.

(3) Debinding: Polymers and lubricants are gradually extracted from green compact at a slow rate of heating in a controlled atmosphere furnace below the degradation temperature of polymers and lubricants through a process called "debinding."

(4) Sintering: Debound AISI 420 martensitic stainless steel parts are sintered in controlled atmosphere furnace between 1300⁰C - 1380⁰C to produce final products.

It has been found that the densities of sintered parts are in between 95 to 98%, almost as high as that of wrought metals. Despite the shrinking which varies from 12-15%, closer and more accurate tolerances can be achieved with injection moulded metals. This is because the plasticity of the feedstock and the nature of the closed injection mold allow greater design freedom than other conventional methods ³⁻⁶. Figure 1 shows the process route for manufacturing of MIM components.

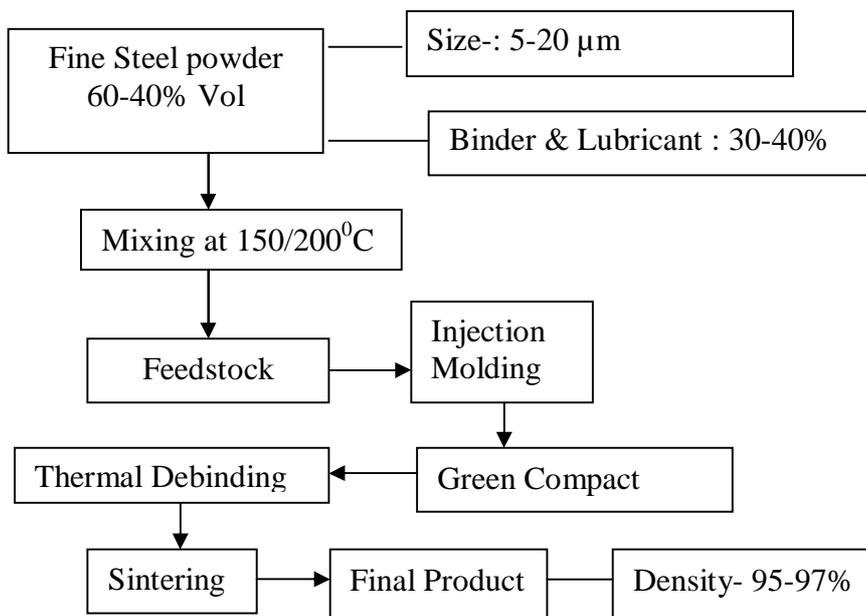


Figure 1: Process route for manufacturing of components by MIM

III. EXPERIMENTAL DETAILS

3.1 Initial raw material

AISI 420 martensitic stainless steel feedstock imported from Advanced Metalworking Inc. Carmel, USA was used for this study. The chemical composition by wt. % of the feedstock is C: 0.55, Cr: 13.3, Mn: 0.3, Si: 0.4, S: 0.005, P: 0.040, Fe: balance. The binder content of the feedstock is 7.0% by weight. Viscosity of the feedstock is about 7980 poise at 175 ⁰C, density is 5.015 gm/cc. and bulk density is 2.0-3.0 gm/cc.

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3.2 Injection Molding

Tensile specimen is chosen as a component for the present investigation. Figure 2 shows the drawing of the tensile specimen as per ASTM standard E-8 with consideration of 15% shrinkage.

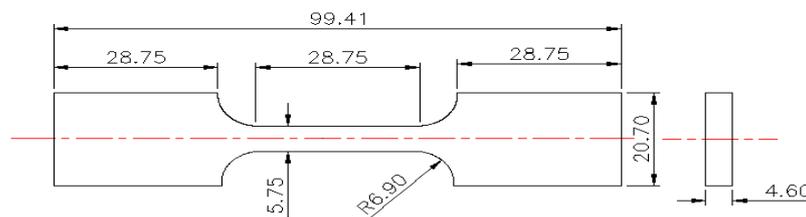


Figure 2: Component geometrical drawing considering 15% shrinkage

The injection temperature and injection pressure of feedstock were optimized by trial and error method using the experimental mold. The selected experimental parameters which were used to prepare green compacts are given in the Table 2.

Table 2: Parameters for Injection molding of AISI 420 feedstock

Product	Material	Injection Pressure psi	Locking Pressure psi	Injection Time sec	Cooling Time sec	Nozzle Temperature °C
Tensile Specimen	AISI 420 feedstock	20	80	10	30	45-50

3.3 Thermal debinding

The samples were placed inside the chamber type of furnace . Debinding was performed in a controlled atmosphere furnace. The continuous flow of N₂ gas (99.9% purity) was maintained inside the furnace to remove the volatile matter of binder. Slow rate of heating from 1-5⁰C/min was maintained to remove slowly the binder material of green compact .The binder material were removed by wicking at lower temperature and by evaporation at higher temperature. The maximum debinding temperature was selected 550⁰C.Total time of debinding was around 6- 6.30 h.

3.4 Sintering

Sintering, the final process in metal injection molding is the final step to produce complex shaped engineering components from powder material. The debound components were placed inside the alumina tube. The alumina tube was connected with a gas cylinder and controlled flow of inert gas (Argon:99.9%) was maintained throughout the sintering cycle to prevent any oxidation. After debinding, components are often near 60% dense, while the final density approaches 96 % or more than 96 % .As soon as the binders are removed at 550⁰C and above, the presintering process starts to develop the bonding of particles.

IV. DESIGN AND EXPERIMENT BASED ON TAGUCHI PARAMETER DESIGN

The objective of the experiments was to determine the effects of sintering parameters on the density of the component and to optimize the set of factors that would maximize the final density. The density was measured by Archimedes principle. Each factor of three sintering factors is designed with three levels as shown in Table 3. This is because the influence of these factors on the result may vary nonlinearly.

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Table 3: Factors and levels for the sintering experiments

Sl. No.	Factor	Level 1	Level 2	Level 3
1	A	1300	1340	1380
2	B	45	60	90
3	C	2	5	10

A – Sintering temperature, °C; B – Sintering time, min; C – Heating rate, °C/min.

An L₉ orthogonal array (OA) was employed in as it was able to provide minimum degrees of freedom required for the experimental design. The column assignment and the experimental layout are shown in Table 4.

Table 4: Experimental layout and factors distribution of L₉ orthogonal array.

Experiment No.	Experimental value					
	A	B	C	Sintering temperature (°C)	Sintering time (min)	Heating Rate (°C min ⁻¹)
1	1	1	1	1300	45	3
2	1	2	2	1300	60	5
3	1	3	3	1300	90	7
4	2	1	2	1340	45	5
5	2	2	3	1340	60	7
6	2	3	1	1340	90	3
7	3	1	3	1380	45	7
8	3	2	1	1380	60	3
9	3	3	2	1380	90	5

Each experiment was conducted with three replications. After the completion of all experiments and the analysis of the data, a confirmatory experiment was performed at the recommended settings. This is an important step in Taguchi's parameter design as it provides indications on the validity of the experimental procedures and results¹⁰⁻¹¹.

Table 5 shows the rate of heating, rate of cooling and holding time at different temperatures that were followed during experiments.

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Table 5: Rate of heating rate of cooling and holding time at different temperatures

Heating	
550 ⁰ C /min -1000 ⁰ C /min	2 ⁰ C /min per minute
1000 ⁰ C /min	Holding time – 60 minutes
1000 ⁰ C-Sintering temperature	Rate of heating
Sintering temperature	Holding time
Cooling	
Sintering Temperature-1000 ⁰ C /min	5 ⁰ C /min per minute
1000 ⁰ C /min	Holding – 60 minutes
1000 ⁰ C /min -200 ⁰ C /min	5 ⁰ C /min per minute
200 ⁰ C /min – room temperature	Furnace cooling

V. EXPERIMENTAL RESULTS

In Taguchi technique, the variation of the response is also examined using an appropriately chosen S/N ratio. Broadly speaking, the S/N ratio is the ratio of the mean (signal) to the standard deviation (noise)¹²⁻¹³. These S/N ratios, derived from the quadratic loss function, are expressed on a decibel (dB) scale. The formula used to compute the S/N ratio depends on the objective function. Generally, three standard S/N equations are widely used to classify the objective function as: ‘larger the better’, ‘smaller the better’, or ‘nominal the best’. However, regardless of the type of performance characteristic, a larger S/N ratio is always desirable.

In the present study, density is a ‘larger the better’ type of quality characteristic since the goal is to maximize the density. Three readings corresponding to the three replications were recorded for each experimental condition as shown in Table 6.

Table 6: Density (gm /cc) of sintered parts and S/N Value

Experiment	R ₁ (gm/cc)	R ₂ (gm/cc)	R ₃ (gm/cc)	Average (gm/cc)	S/N Value
1	7.69	7.22	7.74	7.55	27.11
2	7.29	7.39	7.59	7.42	26.99
3	7.41	7.39	7.44	7.35	26.92
4	7.32	7.2	7.54	7.54	27.14
5	7.63	7.47	7.53	7.19	26.72
6	7.18	7.21	7.18	7.19	26.72
7	7.3	7.37	7.8	7.49	27.08
8	7.79	7.53	7.69	7.67	27.283
9	7.69	7.65	7.69	7.68	27.29

The density of the each sintered sample was measured by Archimedes principle and the results are given in Table 6. The standard S/N ratio computing formula for this type of response is¹³:

$$[S/N]_{LB} = -10 \log \left(\frac{1}{n} \sum_{i=1}^n \frac{1}{y_i^2} \right) \quad (1)$$

where ‘i’ is the number of a trial; ‘Y_i’ is the measured value of quality characteristic for the ith trial; ‘n’ is the number of repetitions for the experimental combination.

Signal-to-noise ratios are computed using Eq. 1 for each of the nine experimental conditions and are reported in Table 6. Since the experimental design is orthogonal, the factor effects can be separated out in terms of the S/N ratio and in terms of the mean response. The average values of S/N ratios of the three control factors at each of the levels are shown in Figure 3, and from which the levels corresponding to the highest S/N ratio values are chosen for each parameter representing the optimum condition.

Here, the optimum condition is corresponding to the maximization of the density. It is clear from Figure 3 that the optimum levels are: A₃ (sintering temperature, °C/min), B₁ (sintering time, min.), C₂ (heating rate, °C/min.) respectively.

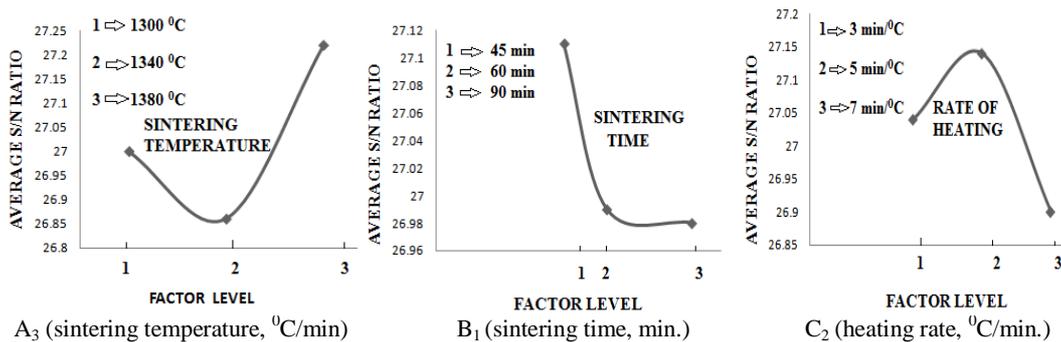


Figure 3: Effects of process parameters on average S/N ratios

In addition to S/N analysis, main effects of the process parameters on the mean response are also analyzed. The mean response refers to the average value of the quality characteristic for each factor at different levels.

Thus the average values of the density for each factor at the three levels have been calculated and are plotted in

Figure 4. The mean response analysis (Figure 4) also indicates the same optimum level of the parameters (A₃, B₁, C₂) as is obtained in S/N ratio analysis.

From the analyses of S/N ratio and the mean response characteristic, the optimum levels of the control factors are determined as: A₃, B₁, and C₂

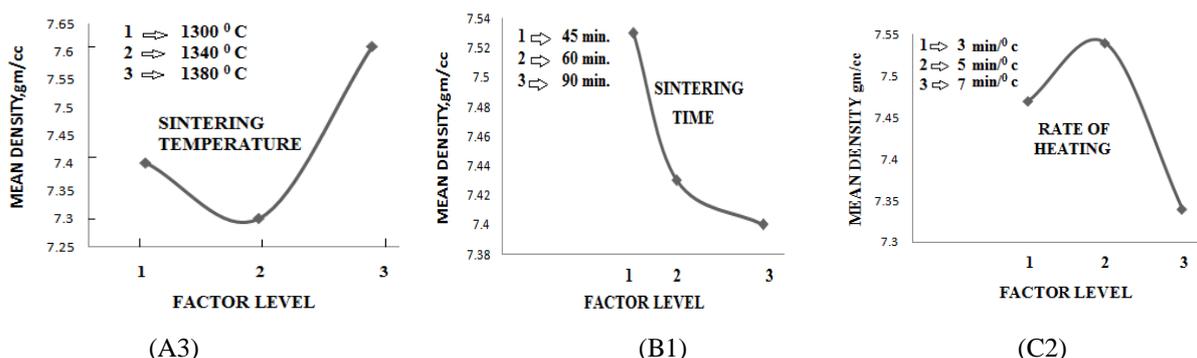


Figure 4: Effects of process parameters on mean response

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5.1. Prediction of optimum quality characteristic (QC)

Hence, the predicted mean of the quality characteristic (density) has been computed as shown by (Chaulia ,Das,2008) the reference [13].

$$D = \text{Density expected result at optimum condition} = T + (A_3 - T) + (B_1 - T) + (C_2 - T) \quad (2)$$

where T is the grand average of performance characteristic [corresponding to all the 27 (= 9 x 3) readings in Table 6]. A₃, B₁, and C₂ are the average values of the density with process parameters at their respective optimal levels and D denotes the predicted mean of the density at optimum condition. The calculated values of various response averages are: T=7.48 gm/cc, A₃=7.61 gm/cc, B₁=7.46 gm/cc, and C₂=7.48 gm/cc. So substituting these in Eq. 2, the mean optimum value of the density has been predicted as: D=7.6 gm/cc.

An important step in Taguchi’s optimization technique is to conduct confirmation experiments for validating the predicted results. As suggested by (Singh, Kumar, 2004) that a confidence interval for the predicted mean on a confirmation run can be calculated using Eqs 3 and 4. Thus a 95% confidence interval (CI) for the predicted mean of optimum QC on a confirmation test is estimated using the following two Eqs 3 and 4 [12]:

$$CI = [F(\alpha, 1, f_e) V_c \{(1/N_{eff}) + (1/R)\}]^{(1/2)} \quad (3)$$

$$N_{eff} = [N / (1 + T_{dof})] \quad (4)$$

where, F(, 1, fe) is the F-ratio required for 100 (1- α) percent confidence interval, fe is DOF for error, Ve is the error variance, R is number of replications for confirmation experiment (= 3), and N_{eff} is effective number of replications. N is total number of experiments [= 27 (9 x 3)] and TDOF is the total degrees of freedom [= 6 (2 x 3)] associated with the estimate of mean optimum. From Table 7, the values are: Ve =0.918, fe= 20, and from standard Statistical Table, the required F-ratio for α= 0.05 is: F(0.05,1,20.) = 4.35. Substituting these values in Eqs 3 and 4, the calculated confidence interval is: CI = ±0.344. Thus the 95% confidence interval of the predicted optimal density is obtained as:

(7.6±0.344) gm/cc
i.e. 7.256 < D(gm/cc) < 7.944

Table 7 : ANOVA for density of sintered parts.

Sl. No.	Factor	DOF	Sum of squares	Variance Mean Square	F Ratio	Remark
1	Sintering temperature	2	0.419	0.2095	7.25	Significant
2	Sintering time	2	0.075	0.038	1.31	Insignificant
3	Heating Rate	2	0.189	0.095	3.28	Insignificant
4	Error (e)	20	0.578	0.0289		
5	Total	26	1.26	0.3885		

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5.2 Confirmation experiment

In order to test the predicted result, confirmation experiment has been conducted by running another three replications at the optimal settings of the process parameters determined from the analysis.

These results are shown in Table 8 and it is observed that the mean density obtained from the confirmation experiments is 7.64 gm/cc, which falls within the predicted 95% confidence interval.

Table 8: Results for confirmation experiments.

Sl. No.		Replication			Average
1	Density (gm/cc)	7.68	7.54	7.63	7.64

VI. CONCLUSION

The final density of AISI 420 martensitic stainless steel MIM specimen was optimized by using Taguchi method. The following conclusions may be drawn from the present investigation work:

- (i) Among the three process parameters, the sintering temperature was having significant effect on the quality characteristic i.e., density.
- (ii) The optimal levels of the process parameters were found to be A_3 , B_1 , and C_2 corresponding to sintering temperature of 1380 °C, sintering time of 45 min, and rate of heating of 5 °C/min. An optimized value of the density for a 95% confidence interval was predicted as:
(7.6 ± 0.344) gm/cc i. e. $7.256 < D \text{ (gm/cc)} < 7.944$
- (iii) From confirmation experiments, the mean value of the density corresponding to the optimum conditions was obtained as 7.64 gm/cc, which fell within the predicted range.
- (iv) Thus the predictions made by Taguchi's parameter design technique were in good agreement with the confirmation results.
- (v) The results of the present investigation were valid within the specified range of the process parameters along with their chosen levels and the feedstock used to manufacture tensile specimen.

REFERENCES

- [1] German, R. M., Powder Injection Moulding, Metal Powder Industries Federation, 1990.
- [2] German, M., Bose .A., Injection Molding of Metals and Ceramics, MPIF, Princeton, 1997.
- [3] Loh . N. H., Khor . K. A., Tor .S. B., March, Injection molding of stainless steel 316L, 2nd A International Symposium on high performance Metal Matrix Composites, Mechanical Engineering Laboratory, Tsukuba City, Japan, pp.5-7,1997.
- [4] Rawers. J., Crooydon. F., Krabbe . R., Duttlinger . N., Powder Metallurgy, 39, 125. 1996.
- [5] Nayar .H. S., Wasiczko. B., Nitrogen absorption control during sintering of stainless steel parts. Metal Powder Report, pp. 611-614, September 1990.
- [6] Cai .L. X., German .R. M., International Journal of Powder Metallurgy, 31 (3). pp.257- 64, 1995.
- [7] Ji .C. H., Loh*. N. H., Khor. K. A., Tor . S. B., Sintering study of 316L stainless steel metal injection molding parts using Taguchi method: final density, Materials Science and Engineering, A311. 74-82. ,2001
- [8] Lance. A . Ealey., Quality by design Taguchi methods™ and US industry, 2nd ed. Sidney Irwin professional publishing and ASI Press, pp. 189-207, 1994.
- [9] Montgomery. D. C., Design and analysis of experiments, John Wiley, 4th ed, 1997.
- [10] Gopalsamy. Bala Murugan, Taguchi method and Anova: An approach for Process parameters optimization of hard machining while machining hardened steel. Journal of Scientific & Industrial Research. Vo.68, 686-695, , August 2009.
- [11] Karna. Shyam Kumar., An overview on Taguchi Method. International Journal of Engineering and Mathematical Sciences. Vol1,11-18, Jan.-June 2012.
- [12] Kmaruddin . S., Application of Taguchi Method in the Optimization of Injection Moulding Parameters for Manufacturing Products from Plastic Blend. IACSIT International Journal of Engineering and Technology. Col2, No.6. ISSN:1793- 8236, December 2010..
- [13] Chaulia . Prabir Kumar., Das. Reeta ., Process Parameter Optimization for Fly Ash Brick by Taguchi Method. Materials Research. Vol.11, No.2, 159-64., 2008.
- [14] Singh .H., Kumar .P., Tool wear optimization in turning operation by Taguchi method. Indian Journal of Engineering and Material Sciences, 11(1), 19-24, 2004.