Process Parameter Selection for Uniform Deposition of ChromeCarbide-NickleChrome (Cr_3C_2-20NiCr) Thermal Flame Spray Coatings on Stainless Steel (SS347H) Boiler tube

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Abstract: - Cr_3C_2−20NiCr coatings were deposited on 347H steel by Thermal Flame spraying. The thickness of the coated surface, cross section of coating and various phase present in coated surface were investigated using various parameter combinations as per Taguchi method in detail. In industries, various types of coatings are used in so many applications. Different methods are employed to protect the material from abrasion, corrosion, degradation, etc. Thermal spraying is one of the most effective methods to protect the material from high temperature corrosion, stresses and erosion, thus increasing the life of material in use. Flame spray coating is a thermal spraying technique known for providing hard, wear resistant and dense microstructures coatings. Scanning Electron Microscopy and X-Ray diffractometer used to analyze uniformity of coating surface and various phase present in coating. In flame spraying process parameters is more affected on the coating performance. By changing the process parameters coating performance is also change. Acetylene pressure, oxygen pressure and distance between coating nozzle and substrate is more affected on coating in flame spray coating. After investigation of coating in SEM, at acetylene pressure at 1.0 bar, oxygen pressure at 4.0 bar and spraying distance at 75 mm give more uniform structure than other combinations of various parameters. The XRD peaks reveal the presence of Cr_3C_2, Cr_7C_3 and Cr_3Ni_2 as main phases. Some medium peaks indicating the presence of CrNi are also revealed by the XRD analysis. The XRD peaks reveal the formation of Cr_7C_3, Cr_3C_2 as strong phases and CrNi, Cr_2C and Cr as the medium intensity phases and Ni as a weak intensity phase in as-sprayed 347H steel.

Keywords: Thermal flame spraying; Cr_3C_2-NiCr; ANOVA; Uniformity of thickness.

1 Introduction

Cr3C2-NiCr compound coating, which is widely used in aerospace, metallurgy and electric power field, has excellent property of wear resistance, oxidation resistance and corrosion resistance at high temperature. The coating can be prepared by HVOF spraying process, arc spraying process, plasma spraying process or thermal flame spraying process. Cr_3C_2 compound coating prepared with arc spraying process have lower bond strength and higher porosity than that made by the other three processes. It is mainly used for boiler tube protection in power industry and is not suitable for the application of higher mechanics function's request but thermal spraying process is a one of the best choice for it. [1] Cr_3C_2-NiCr compound coating prepared with HVOF spraying process has the highest bond strength and density, which is mainly used in aerospace field. Now many kinds of ceramic coatings are prepared by thermal flame spraying process spraying process because of their high melting point. Flame spraying process gives same performance as a HVOF spraying process with minimum noise and minimum use of gases. Ceramic powder melts well during thermal spraying and this helps to enhance the quality of the coating. In this article thermal flame spraying process was studied to prepare Cr_3C_2-NiCr coating. [2] The uniformity of coating was tested in detailed at various process parameters combinations as per taguchi method.

2 Experimental

2.1 Material and method

2.1.1. Substrate material

Stainless Steel 347 has slightly better corrosion resistance than type 321 stainless steel in strongly oxidizing environments. Alloy 347 is a columbium (niobium)/tantalum stabilized austenitic chromium-nickel stainless steel which was developed to provide an 188 type alloy with improved intergranular corrosion resistance. [3] Alloy 347 has a slightly improved corrosion resistance over other alloys of stainless steel in strongly oxidizing environments as a result of the addition of columbium (niobium) and tantalum. Columbium tantalum carbides precipitate within the grains instead of forming at the grain boundaries. Type 347 should be considered for applications requiring intermittent heating between 800ºF (427ºC) and 1650ºF (899ºC), or for welding under conditions which prevent a post weld anneal.

2.1.2. Coating material

When compared to coating materials containing tungsten carbide, coating materials that contain chromium carbide can withstand higher service temperatures up to 870 °C (1600 °F). When applied using the HVOF spray process, coatings of these materials are very dense, have very good bond strength and are more homogeneous compared to plasma or combustion powder thermal spray coatings.The coating material is Cr_3C_2-20%NiCr compound powder, which contains 20% NiCr alloy and 80% Cr_3C_2. [3] The size of the Cr_3C_2-20%NiCr in powder form is between 10~30µm. Composition of coating wire shown in table 1.

Table.1 Spraying coating wire characteristics.

Coating wire type	Chemical composition (wt.%)				Apparent density (gm/cm^3)
	Ni	C	O	Cr	
Cr_3C_2-20%NiCr	21.1	8.9	0.3	Bal.	2.5–3.2

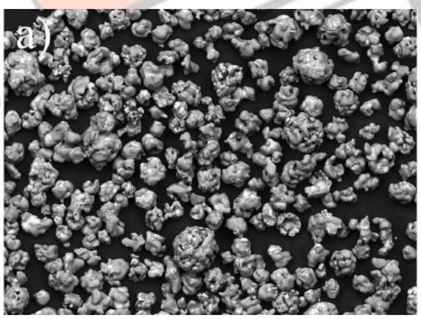


Fig. 1. SEM micrographs of the powders Cr_3C_2–NiCr

2.3. Thermal spraying experiments

Thermal flame spraying system (MASTER JET@ Rokide Flame spray gun) with standard configuration was utilized for grit blasting the substrates (nozzle, combustion chamber, short axial wire injector), and utilizing Cr_3C_2-20%NiCr and acetylene and oxygen as a combustion gas. Cr_3C_2–NiCr were sprayed using the flame spray gun used for grit blasting the substrates. [4] Range of process parameters for flame spray coating as shown in table 2.

Table.2 Range of Thermal flame spraying parameters

Coating wire type	Oxygen pressure in bar	Acetylene pressure in bar	Wire feeding rate in mm/sec	spraying distance (mm)	Oxygen flow in lpm	Acetylene flow in lpm
Cr_3C_2-20%NiCr	3.5 to 4.5	1.0 to 1.2	0.31mm/sec	50 to 100	80	70

2.4 Taguchi method and ANOVA

Dr. Taguchi of Nippon Telephones and Telegraph Company, Japan has developed a method based on "ORTHOGONAL ARRAY" experiments which gives much reduced "variance" for the experiment with "optimum settings "of control parameters. Thus, the combination of Design of Experiments with optimization of control parameters to obtain BEST results is achieved in the Taguchi Method. [5]ANOVA is used in the analysis of comparative experiments, those in which only the difference in outcomes is of interest. The statistical significance of the experiment is determined by a ratio of two variances. This ratio is independent of several possible alterations to the experimental observations: Adding a constant to all observations does not alter significance. Multiplying all observations by a constant does not alter significance. So ANOVA statistical significance result is independent of constant bias and scaling errors as well as the units used in expressing observations.

2.5 Characterization of coatings

Cross-sections of the coating were metalographically prepared for uniformity of coated surface evaluations. The morphology of the specimens was characterized by a QUANTA 200 FEG scanning electron microscope (SEM). The phases presented at the more uniform coated surface was identified by X-ray diffraction (XRD) using X'Pert PRO diffractometer with Cu K_α (α = 0.154 nm) radiation in the diffraction angle between 25◦ and 85◦.

3. Results and discussion

3.1 Taguchi method for design of experiments

3.1.1 Selection of the thermal flame spray parameters and their levels

Acetylene pressure and oxygen pressure are more effective on heat generation in flame spray coating process and thereby uniform structure and thickness of coating are achieved. Nozzle distance from substrate also affects on velocity of partical and oxidation due to interaction with environment between nozzle and substrate. Selected parameters are presented in Table 5.1.

Table. 3 Levels of parameters for DOE

Sr.No.	PARAMETERS	Level 1	Level 2	Level 3
1	Acetylene Pressure (bar)	1.0 bar	1.1 bar	1.2 bar
2	Oxygen Pressure (bar)	3.5 bar	4.0 bar	4.5 bar
3	Nozzle distance from substrate material (mm)	50 mm	75 mm	100 mm

3.1.2 Selection of orthogonal array

Orthogonal array is used for reduce the experiments. By using full fractional or partial fraction method orthogonal array is selected. [6] By using two factorial design experiment can reduce significantly.

Here three level and three parameters are selected so instead of 3^3 = 27 only 3^(4-2) = 9 experiments are run. So L_9 orthogonal array is selected. The experimental layout for the flame spray coating parameters using L_9 orthogonal array is shown in table 4.

Table. 4 Orthogonal array Coded(L_9)

SR. NO.	Acetylene Pressure in BAR	Oxygen Pressure in BAR	Nozzle distance from substrate material in mm
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

3.1.3 Procedure for measuring thickness of coating surface

DM5E is ergonomically designed with a weight of just 223g, including its AA batteries, which allow up to 60 hours of operation. The basic version is specified to EN 15317 and features an LCD data display, which is backlit to be visible in all lighting conditions. Instrument operation is carried out with one hand via a user-friendly interface.After performing coating operation according to taguchi design, on all response as a thickness to be measured by thickness gauge as shown in figure 2 and results also display in table 5

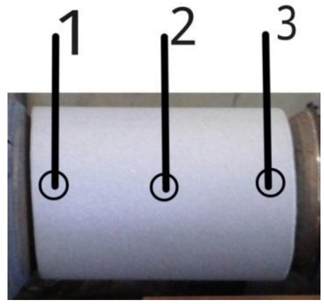


Fig. 2 Thickness measurements at various points

Table 5. Results of DOE

Run	Acetylene Pressure in BAR (A)	Oxygen Pressure in BAR (B)	Nozzle distance from substrate material in mm (C)	Average coating thickness in mm (Response)
1	1.0	3.5	50	0.265
2	1.0	4.0	75	0.231
3	1.0	4.5	100	0.261
4	1.1	3.5	75	0.262
5	1.1	4.0	100	0.221
6	1.1	4.5	50	0.275
7	1.2	3.5	100	0.268
8	1.2	4.0	50	0.255
9	1.2	4.5	75	0.281

3.2 Analysis of variance (ANOVA)

The purpose of Analysis of variance (ANOVA) is to find which parameters significantly affected the quality characteristic. ANOVA generalizes to the study of the effects of multiple factors. [7] When the experiment includes observations at all combinations of levels of each factor, it is termed factorial. Factorial experiments are more efficient than a series of single factor experiments and the efficiency grows as the number of factors increases. Consequently, factorial designs are heavily used.

Table 6. Analysis of variance table [Classical sum of squares - Type II]

Source	Sum of Square	Df	Mean Squares	F Value	p-value Prob> F
Model	3.433E-003	6	5.129E-004	**48.63**	0.0199 significant
A-Acetylene Pressure	4.807E-004	2	2.403E-004	**23.26**	0.0412
B-Oxygen pressure	2.559E-003	2	1.129E-003	**109.29**	0.0091
C-Distance from nozzle to substrate	3.380E-004	2	1.690E-004	**16.35**	0.0576
Residual	2.067E-004	2	1.033E-005		
Cor total	3.098E-003	8			

The Model F-value of 49.63 implies the model is significant. There is only a 1.99% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob> F" less than 0.0500 indicate model terms are significant. In this case A, B are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

Std. Dev.	3.215E-003	R-Squared	0.9933
Mean	0.26	Adj R-Squared	0.9733
C.V. %	1.25	Pred R-Squared	0.8649
PRESS	4.185E-004	Adeq Precision	21.164

The "Pred R-Squared" of 0.8649 is in reasonable agreement with the "Adj R-Squared" of 0.9733. "Adeq Precision" measures the signal to noise ratio. A ratio greater than 4 is desirable. Your ratio of 21.164 indicates an adequate signal. This model can be used to navigate the design space.

Table 7. Coefficient of estimate

Term	**Coefficient Estimate**	**Df**	**Standard Error**	**95% CI Low**	**95% CI High**
Intercept	0.26	1	1.072E-003	0.25	0.26
A[1]	-5.333E-003	1	1. 515E-003	-0.012	1.187E-003
A[2]	-5.000E-003	1	1.515E-003	-0.012	1.520E-003
B[1]	7.333E-003	1	1.515E-003	8.133E-004	0.014
B[2]	-0.022	1	1.515E-003	0.029	-0.015
C[1]	7.333E-003	1	1.515E-003	8.133E-004	0.014
C[2]	3.333E-004	1	1.515E-003	-6.187E-003	6.853E-003

3.2.1 One-way ANOVA analysis

3.2.1.1 Acetylene pressure v/s thickness, Oxygen pressure v/s thickness and Nozzle to substrate distance v/s thickness

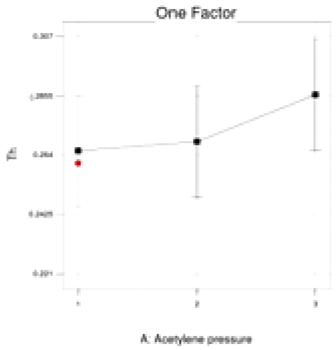


Fig. 3 Acetylene pressure v/s thickness

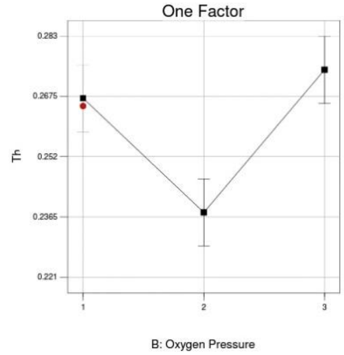


Fig. 4 Oxygen pressure vs thickness

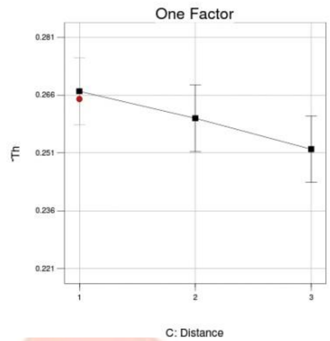


Fig. 5Nozzle to substrate distance v/s thickness

According to fig. 3, 4, and 5 we can say that, thickness of the coating increase with increase in acetylene pressure, thickness of the coating decreases with increase in oxygen pressure at some level but further increase in oxygen pressure thickness is increase and thickness of the coating decrease with increase in acetylene pressure.

3.2.1.2 Normal plot of residuals and Residuals vs. Predicted

Further, adequacy of developed model has been checked from the normal probability plot and residuals vs. predicted response plot for the coating thickness. Fig.6 and Fig.7 show the normal percentage probability plot of the internally studentized residuals and plot of the residuals versus the predicted response for the quadratic cutting force model. It is observed from these plots that the residuals fall on a straight line in normal plot of residuals (Figure 6) implying that the errors are distributed normally and there is no obvious pattern and unusual structure in residuals versus predicted plot (Figure 7). This implies that the model proposed is adequate and there is no reason to suspect any violation of the independent or constant variance assumption. Hence, the model can be used for further analysis to determine the effects of various process parameters on the response.

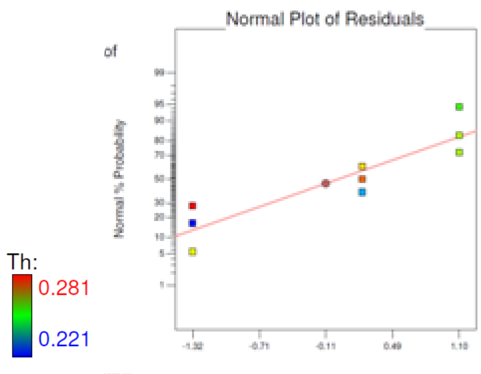


Fig. 6 Normal plot of residuals

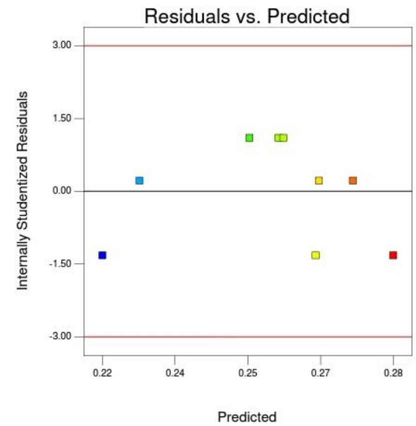


Fig. 7 Residuals vs. Predicted

3.3 Scanning electron microscopy and analysis of coating uniformity

SEM images of different sample and their parameter are shown in fig. 8 to 15. According to various parameters and their effects on coating uniformity are easily analyzed by SEM images.

From above SEM images it can be conclude that flame spray coating parameters significantly effect on coating structure uniformity. From fig. 9, 12 and 15 we can analyze that oxygen pressure at 4.0 bar give more uniform structure than other oxygen pressure value. For combination of other parameter, coating structure is less uniform.

It can be also said that acetylene pressure at 1.0 bar and spraying distance at 75 mm give more uniform structure than other combinations of various parameters as shown in fig. 10 to 16.

Porosity between coating structure and substrate is also observed from SEM images as shown in fig 12. For parameters, Acetylene pressure: 1.1 bar, Oxygen pressure: 4.0 bar, Spray distance: 100mm porosity is less compare to combinations of other parameters.

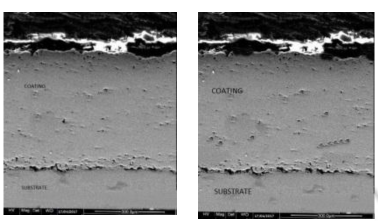


Fig. 8 SEM images for cross section of coating at a) Acetylene pressure : 1.0 bar b) Oxygen pressure : 3.5 bar c) Spray distance : 50mm.

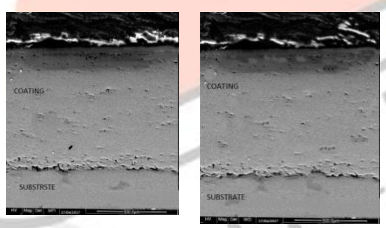


Fig. 9 SEM images for cross section of coating at a) Acetylene pressure : 1.0 bar b) Oxygen pressure : 4.0 bar c) Spray distance : 75mm.

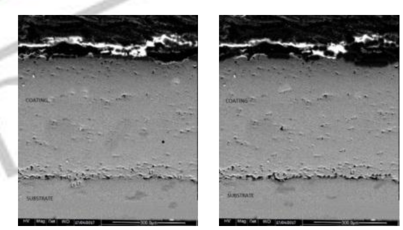


Fig. 10 SEM images for cross section of coating at a) Acetylene pressure : 1.0 bar b) Oxygen pressure : 4.5 bar c) Spray distance : 100mm.

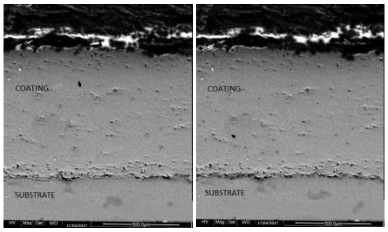


Fig. 11 SEM images for cross section of coating at a) Acetylene pressure : 1.1 bar b) Oxygen pressure : 3.5 bar c) Spray distance : 75mm.

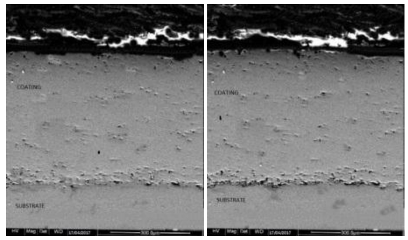


Fig. 12 SEM images for cross section of coating at a) Acetylene pressure : 1.1 bar b) Oxygen pressure : 4.0 bar c) Spray distance : 100mm.

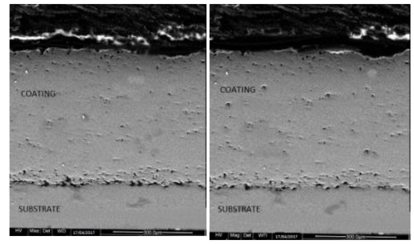


Fig. 13 SEM images for cross section of coating at a) Acetylene pressure : 1.1 bar b) Oxygen pressure : 4.5 bar c) Spray distance : 50mm.

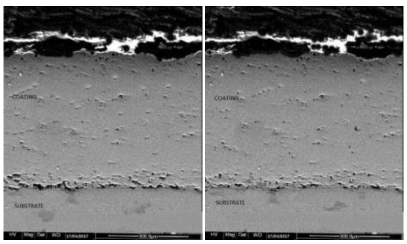


Fig. 14 SEM images for cross section of coating at a) Acetylene pressure : 1.2 bar b) Oxygen pressure : 3.5 bar c) Spray distance : 100mm.

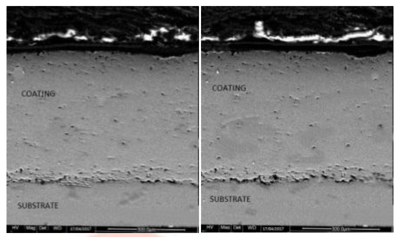


Fig. 15 SEM images for cross section of coating at a) Acetylene pressure : 1.2 bar b) Oxygen pressure : 4.0 bar c) Spray distance : 50mm.

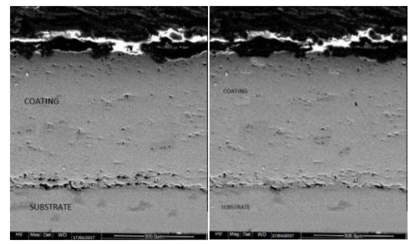
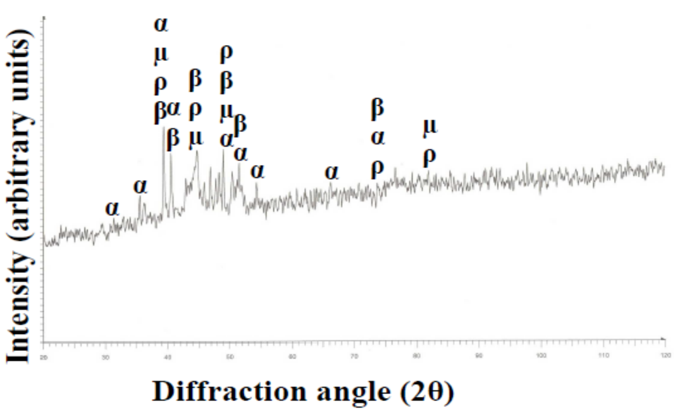


Fig. 16 SEM images for cross section of coating at a) Acetylene pressure : 1.2 bar b) Oxygen pressure :4.5 bar c) Spray distance : 75mm.

3.4 XRD analysis of selected sample

The XRD analysis of the flame spray Cr_3C-NiCr coated 347H boiler steel is compiled in fig. 17. The XRD peaks reveal the presence of Cr_3C_2, Cr_7C_3 and Cr_3Ni_2 as main phases. Some medium peaks indicating the presence of CrNi are also revealed by the XRD analysis. The XRD peaks reveal the formation of Cr_7C_3, Cr_3C_2 as strong phases and CrNi, Cr_2C and Cr as the medium intensity phases and Ni as a weak intensity phase in as-sprayed 347H steel.



Phases	ICDD Number
α-Cr3C2	14-406
μ-Cr3Ni2	26-430
β-Cr7C3	11-550
ρ-CrNi	26-429

Fig. 17 X-ray diffraction pattern for the flame spray Cr_3C_2-NiCr coated 347H steel tube

4 Conclusions

Thermal flame spray coating gives same performance as other popular methods which are used in industries. Flame spray coating gun used wire as a coating material, so deposition of unmelted partical in coated surface is largely reduced. By using Taguchi method it is conclude that process parameters are more affected on coating performance like uniformity of coating surface. For flame spray coating it is need to optimized more affected coating parameter likes a) Acetylene pressure, b) Oxygen pressure and c) Distance between coating gun nozzle and substrate. Taguchi method is largely reduced the number of experiment performed for the coating experiments and give best combination for process parameters. ANOVA results show that by increases in acetylene pressure coating thickness is also an increase because more heat is generated due to more gas pressure. Coating thickness is decreases with increases in distance between nozzle and substrate because with increase in distance impact of partical on substrate is less so coating structure is more porous. Spray distance is also effect on coating thickness. As per taguchi method nine run of experiment performed on different specimen and its SEM images used to analyze surface uniformity of coated surfaces. It can be also said that acetylene pressure at 1.0 bar, Oxygen pressure at 4.0 bar and spraying distance at 75 mm give more uniform structure than other combinations of various parameters for Taguchi experiment. The XRD peaks also reveal the formation of Cr_7C_3, Cr_3C_2 as strong phases and CrNi, Cr_2C and Cr as the medium intensity phases and Ni as a weak intensity phase in as-sprayed 347H steel.

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