




# Pack Siliconizing Optimization of AISI D2 Tool Steel

Mojtaba Najafizadeh<sup>1</sup> · Morteza Hosseinzadeh<sup>2</sup> · Mehran Ghasempour-Mouziraji<sup>3,4</sup> · Angelo Perrone<sup>5</sup> · Pasquale Cavaliere<sup>5</sup> 

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## Abstract

The paper describes the pack siliconizing optimization procedure for AISI D2 tool steel. Pack siliconizing was performed by employing different treating temperatures (falling in the range 650–950 °C) and times (falling in the range 2–4 h). The siliconizing atmosphere was obtained through silicon (at 12 wt.%) with the addition of different percentages of NH<sub>4</sub>Cl (falling in the range 0.5–1 wt.%), Al<sub>2</sub>O<sub>3</sub> was used as halide activator. The coatings' phases evolution was analyzed through X-ray diffraction; the coatings' thickness was measured through scanning electron microscopy observations of the cross section of the samples; the mechanical properties were evaluated through micro hardness measurements. The results showed that the coatings' thickness and hardness increase as the treating time and temperature as well as the halide activator percentage increase. The coating procedure optimization was performed by employing the response surface methodology based on Box-Behnken method. The results of the optimization procedure led to the of the optimal combination of processing parameters: 1 wt.% of halide activator, 3.575 holding time, 950 °C in temperature.

**Keywords** Siliconizing · Coatings · Multi-objective optimization · Box-Behnken · Mechanical property

✉ Pasquale Cavaliere  
pasquale.cavaliere@unisalento.it

Mojtaba Najafizadeh  
mojtaba@sjtu.edu.cn

Morteza Hosseinzadeh  
m\_hosseinzadeh59@yahoo.com

Mehran Ghasempour-Mouziraji  
mehran@ua.pt

Angelo Perrone  
angelo.perrone@unisalento.it

- <sup>1</sup> The State Key Laboratory of Metal Matrix Composites, School of Materials Science and Engineering, Shanghai Jiao Tong University, Shanghai 200240, China
- <sup>2</sup> Department of Mechanical Engineering, Ayatolla Amoli Branch-Islamic Azad University, Amol, Iran
- <sup>3</sup> TEMA - Centre for Mechanical Technology and Automation, University of Aveiro, Aveiro, Portugal
- <sup>4</sup> EMaRT Group, School of Design, Management and Production Technologies, University of Aveiro, Estrada do Cercal 449, 3720-509 Oliveira de Azeméis, Portugal
- <sup>5</sup> Department of Innovation Engineering, University of Salento, Via per Arnesano, 73100 Lecce, Italy

## 1 Introduction

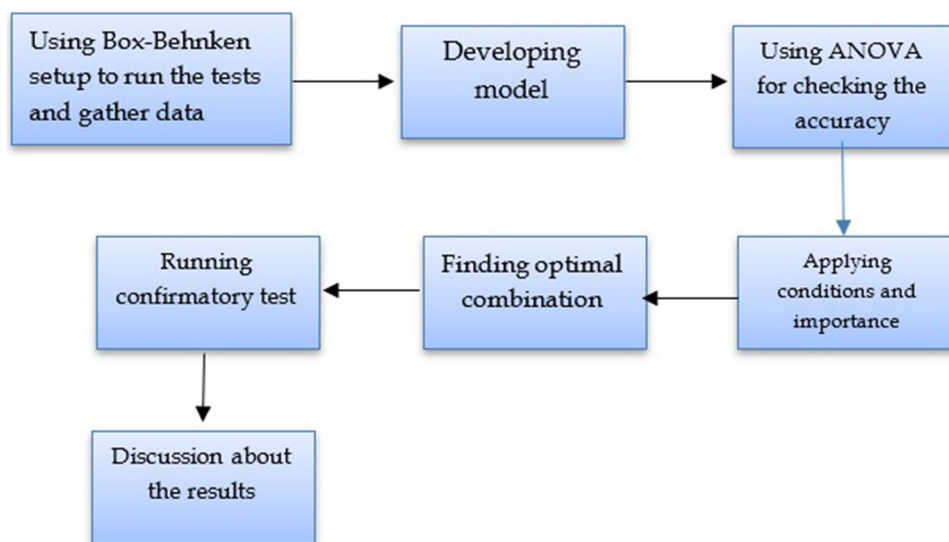
D-type tool steels are high carbon and high chromium ferrous alloys. These steels are characterized by the retention of high strength levels as the working temperature increases. Other fundamental properties are high corrosion and wear resistances [1, 2]. For these reasons, the D2 type steel is mainly employed for the construction of cold extrusion dies [3, 4].

The coating procedures for metals and alloys compounds are performed in order to increase surface mechanical and chemical-physical properties by retaining the bulk characteristics (in particular ductility). The main available coating technologies for tool steels are Chemical Vapor Deposition (CVD), Physical Vapor Deposition (PVD) and Thermal Spray [5]. The Halide Activator Pack Cementation (HAPC) is a process similar to CVD [6]. HAPC is useful to increase the surface properties in a very effective way [7]. In addition, it is widely applied for the coating of large size and very complex shape steel components [8]. Among the HAPC available methods, pack cementation results very versatile with low production costs [9].

**Table 1** Employed processing parameters

| Factors          | Symbol    | Unit         | Actual value- Level 1 | Actual value- Level 2 | Actual value- Level 3 |
|------------------|-----------|--------------|-----------------------|-----------------------|-----------------------|
| Time             | <i>T</i>  | <i>hour</i>  | 2-(−1)                | 3-(0)                 | 4-(1)                 |
| Halide activator | <i>H</i>  | <i>wt.%</i>  | 0.5-(−1)              | 0.75-(0)              | 1-(1)                 |
| temperature      | <i>Te</i> | <i>Deg C</i> | 650-(−1)              | 800-(0)               | 950-(1)               |

**Fig. 1** schematic view of optimisation process



During pack cementation, cementing powders and filler materials mixing to completely cover the steel to be treated are employed. The activator allows for the reactions development on the steel surface in order to induce the hardening compounds precipitation on different coating layers [10]. The thermo-chemical coating method is performed at high temperature (generally in the range 800–1000 °C) for various times (generally in the range 2–10 h) depending on the steel type. One of the main limits of this coating method is the time consuming. This can lead to excessive grain growth with consequent drop of the mechanical properties [11].

So, the main goal of pack siliconizing optimization is the reduction of treating time and temperature by retaining acceptable levels of surface hardness and hardening compounds penetration. For this reason, the optimization of processing parameters is fundamental for the development of pack cementation in general and siliconizing in particular [12–17]. The main aim of the present paper is the obtaining of the best combination of processing parameters basing on experimental results and running

**Table 2** Inputs and outputs value

| Run | A:Halide | B:Time | C:Temperature | Thickness | Hardness |
|-----|----------|--------|---------------|-----------|----------|
| 1   | 0.75     | 4      | 650           | 89        | 778      |
| 2   | 1        | 2      | 800           | 51        | 768      |
| 3   | 1        | 4      | 800           | 92        | 774      |
| 4   | 0.5      | 3      | 650           | 47        | 765      |
| 5   | 0.75     | 2      | 650           | 24        | 735      |
| 6   | 0.5      | 3      | 950           | 143       | 781      |
| 7   | 0.75     | 4      | 950           | 181       | 792      |
| 8   | 0.5      | 2      | 800           | 30        | 746      |
| 9   | 0.75     | 3      | 800           | 38        | 758      |
| 10  | 0.5      | 4      | 800           | 56        | 769      |
| 11  | 0.75     | 3      | 800           | 38        | 758      |
| 12  | 0.75     | 3      | 800           | 38        | 758      |
| 13  | 1        | 3      | 950           | 356       | 800      |
| 14  | 0.75     | 3      | 800           | 38        | 758      |
| 15  | 1        | 3      | 650           | 54        | 763      |
| 16  | 0.75     | 2      | 950           | 168       | 786      |
| 17  | 0.75     | 3      | 800           | 38        | 758      |

**Table 3** ANOVA for thickness

| Source         | Sum of Squares | df | Mean Square | F-value | p value |             |
|----------------|----------------|----|-------------|---------|---------|-------------|
| Model          | 1.085E+05      | 9  | 12,052.78   | 12.75   | 0.0014  | significant |
| A-A            | 9591.12        | 1  | 9591.12     | 10.15   | 0.0154  |             |
| B-B            | 2628.13        | 1  | 2628.13     | 2.78    | 0.1394  |             |
| C-C            | 50,244.50      | 1  | 50,244.50   | 53.15   | 0.0002  |             |
| AB             | 56.25          | 1  | 56.25       | 0.0595  | 0.8143  |             |
| AC             | 10,609.00      | 1  | 10,609.00   | 11.22   | 0.0122  |             |
| BC             | 676.00         | 1  | 676.00      | 0.7152  | 0.4257  |             |
| A <sup>2</sup> | 3041.12        | 1  | 3041.12     | 3.22    | 0.1160  |             |
| B <sup>2</sup> | 244.80         | 1  | 244.80      | 0.2590  | 0.6265  |             |
| C <sup>2</sup> | 30,510.59      | 1  | 30,510.59   | 32.28   | 0.0007  |             |
| Residual       | 6616.75        | 7  | 945.25      |         |         |             |
| Lack of Fit    | 6616.75        | 3  | 2205.58     |         |         |             |
| Pure Error     | 0.0000         | 4  | 0.0000      |         |         |             |
| Cor Total      | 1.151E+05      | 16 |             |         |         |             |

**Table 4** ANOVA for hardness

| Source         | Sum of Squares | df | Mean Square | F-value | p value  |             |
|----------------|----------------|----|-------------|---------|----------|-------------|
| Model          | 4251.74        | 9  | 472.42      | 41.08   | < 0.0001 | significant |
| A-A            | 242.00         | 1  | 242.00      | 21.04   | 0.0025   |             |
| B-B            | 760.50         | 1  | 760.50      | 66.13   | < 0.0001 |             |
| C-C            | 1740.50        | 1  | 1740.50     | 151.35  | < 0.0001 |             |
| AB             | 72.25          | 1  | 72.25       | 6.28    | 0.0406   |             |
| AC             | 110.25         | 1  | 110.25      | 9.59    | 0.0174   |             |
| BC             | 342.25         | 1  | 342.25      | 29.76   | 0.0010   |             |
| A <sup>2</sup> | 121.64         | 1  | 121.64      | 10.58   | 0.0140   |             |
| B <sup>2</sup> | 3.22           | 1  | 3.22        | 0.2803  | 0.6129   |             |
| C <sup>2</sup> | 810.59         | 1  | 810.59      | 70.49   | < 0.0001 |             |
| Residual       | 80.50          | 7  | 11.50       |         |          |             |
| Lack of Fit    | 80.50          | 3  | 26.83       |         |          |             |
| Pure Error     | 0.0000         | 4  | 0.0000      |         |          |             |
| Cor Total      | 4332.24        | 16 |             |         |          |             |

optimization procedure in the following order. Firstly, to gather data in the standard way, Box-Behnken method is used; then by using response surface methodology (RSM) empirical model is introduced. Box-Behnken is an experimental design for RSM. In this experimental

design, each independent variable has three equal spaces values coded as  $-1, 0, 1$ . In this design based on the number of experiments, some tests are repeated to check the accuracy of the achieved results and input

values. Also, in this method, a quadratic model is used as process.

Next, by using an analysis of variance, the accuracy of the model is validated. At this stage, the best combination of parameters based on the boundary conditions is defined. Afterward, the confirmatory test is needed to verify the achieved results. Having had all of data, the complementary discussion on the achieved results is presented.

## 2 Experimental Procedure

The employed bulk was AISI D2 steel with the following composition (in wt.% measured through spectrometry): Fe-1.43, C-0.198, Si-0.308, Mn-0.012, P-0.0075, S-12, Cr-0.8, Mo-0.0304, Ni-0.0901, Cu-0.124 V.

For the pack siliconizing procedure, three different powder compositions were employed. The powders contained different amounts of halide activator  $\text{NH}_4\text{Cl}$  (0.5, 0.75 and 1 wt.%) with the same percentage of Si (12 wt.%).  $\text{Al}_2\text{O}_3$  was used as filler. Silicon is the coating base material,  $\text{NH}_4\text{Cl}$  is added as chemical activator, and  $\text{Al}_2\text{O}_3$  is added as filler.

Roads of D2 tool steel were cut into the form of cylindrical samples with the dimensions of 30\*30 mm. Then, they were polished with the SiC papers up to 1200 finishing. Then, the specimens were cleaned for the coating operation. The cubic stainless steel boxes (with the dimension of the 10x10x10 cm<sup>3</sup>) were fabricated for filling up the half of the box with powder mixture. After the positioning on the powders, the AISI D2 specimens were covered with other powders up to filling the box. The boxes were heated inside a furnace in environment atmosphere (Hefei Kejing Material Technology Company, model KSL-1400X) at different temperatures (650, 800 and 950 °C) for 2, 3 and, 4 h.

After coating operations, the samples were cut and prepared through standard metallographic techniques. The microstructure was characterized by employing a ZEISS EVO 40 scanning electron microscope. The coatings' phases were analysed through X-Ray diffraction with a Rigaku diffractometer at an accelerating voltage of 40 kV and a current of 40 mA with a scan step of 0.02. The micro-hardness was measured by using a diamond indenter with 100 g maximum load.

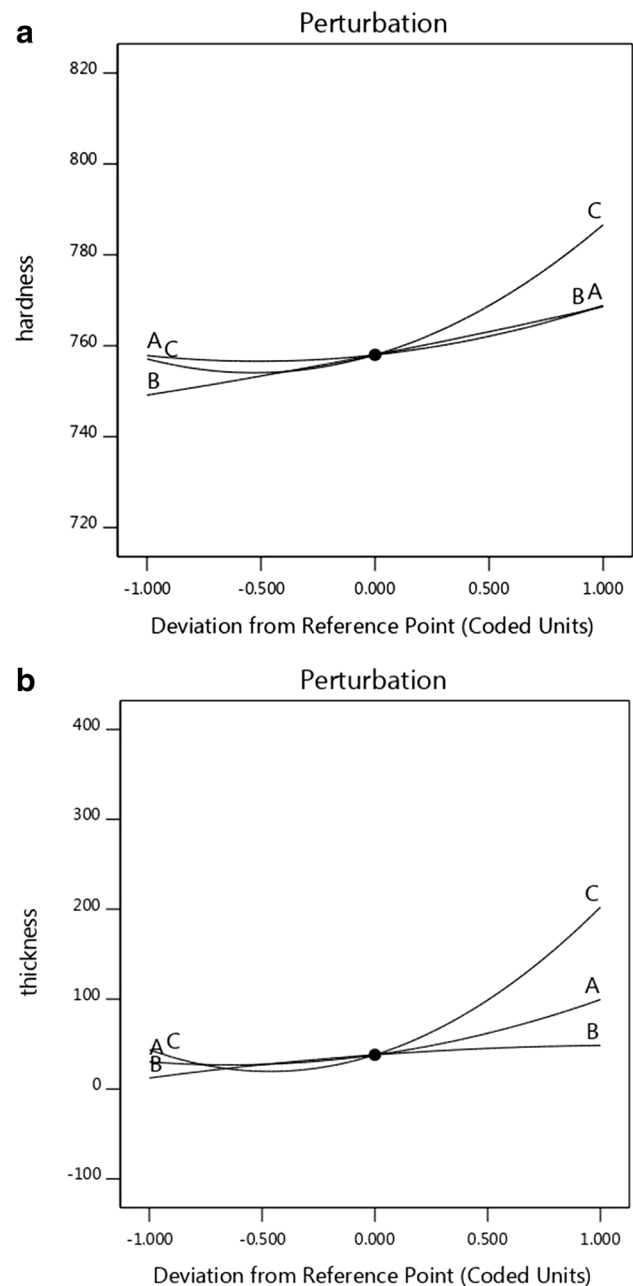
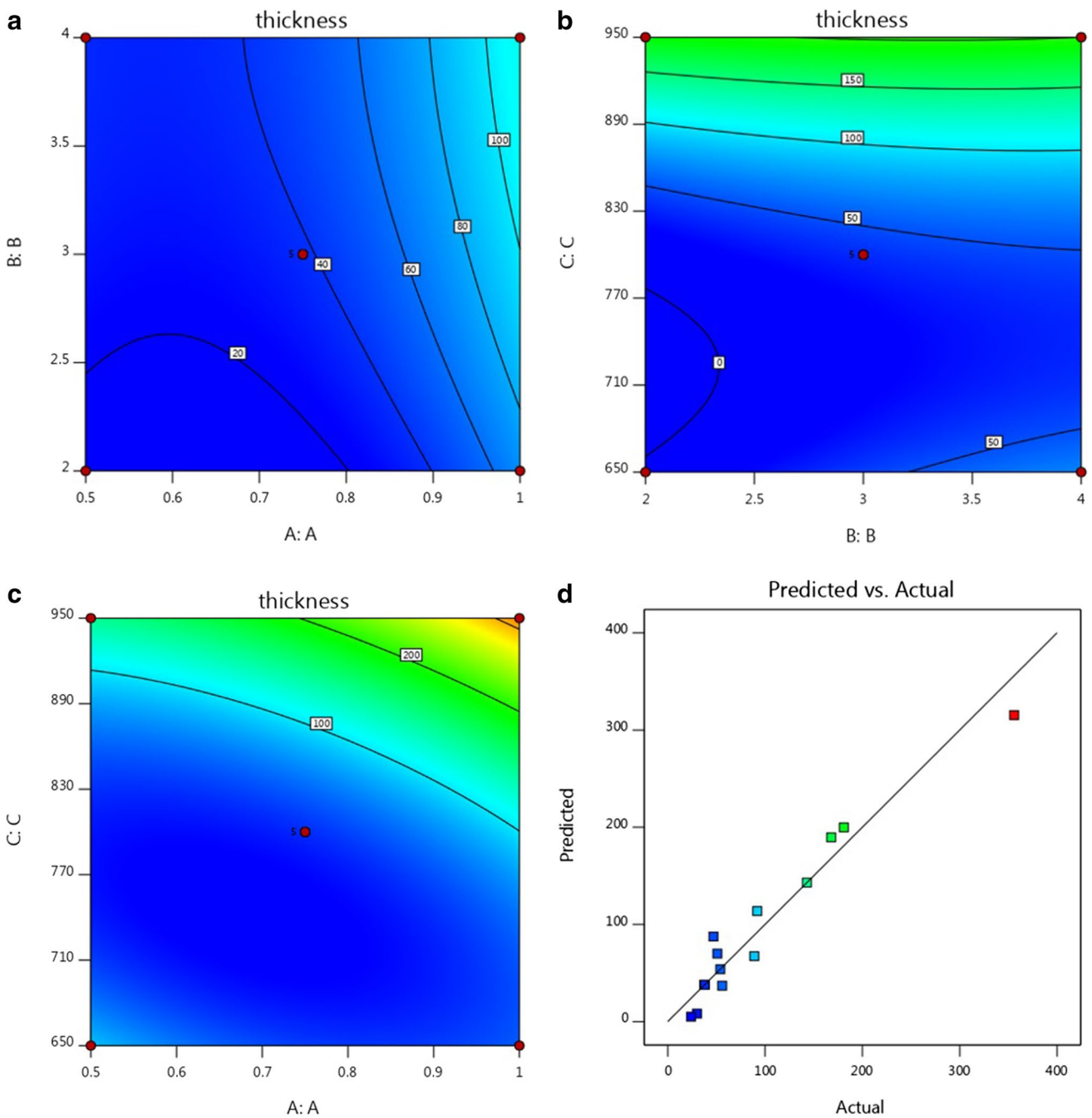


Fig. 2 Perturbation graph for the employed parameters



**Fig. 3** a-d Interaction between temperature and halide, b Interaction time and halide, c Interaction between halide and temperature, d predicted and actual results

### 3 Optimization Procedure

#### 3.1 Design Matrix Creation for Response Surface Method

Hardness and thickness are the main outputs of current study which were considered to evaluate the best

combination of the mentioned parameters. 17 standardized tests were run based on the Box-Behnken approach. RSM is an effective technique to reach the best combination of parameters during coating operations [18]. By using a design of experiment (DOE) approach in Design Expert software, experimental tests

were performed and the results were inserted into the software for the next step.

Halide activator percentage, temperature and time are the input for the current study as listed in Table 1. Figure 1 shows the schematic view of the optimisation procedure.

In the RSM, the distance between the variables must be equal and the lowest, middle and highest values are indicated as (−1), (0) and (1) respectively. The used Design matrix and responses value are shown in Table 2. 5 tests were repeated in order to validate the accuracy of the results.

At this stage, after sorting the results, the empirical equations for outputs are achieved from Design Expert which shows the relationship between input and outputs.

$$\text{Thickness} = +2701.41667 - 1650.16667 A + 121.95833 \text{ time} - 6.29500 C + 15.00000 A * B + 1.37333 A * C - 0.086667 B * C + 430.00000 \text{ holide}^2 - 7.62500 B^2 + 0.003783 C^2 \quad (1)$$

$$\text{Hardness} = +982.25000 - 168.00000 A + 66.58333 B - 0.808333 C - 17.00000 A * B + 0.140000 A * C - 0.061667 B * C + 86.00000 A^2 + 0.875000 B^2 + 0.000617 C^2 \quad (2)$$

#### 4 Effect of Process Parameters on the Thickness Distribution

Figure 3a to c show the three effective parameters leading to the variation of the thickness of the coatings' surface. The thickness distribution of the silicide coating significantly increases with the addition 0.5 to 1 wt.% of the halide activator to the powder compounds. Also, the other effective factors for increasing the thickness of the silicide layer are treatment time and temperature. With increasing the percentage of halide activator, treatment time and temperature the thickness of the silicide coating significantly enhances, as shown in Fig. 3. The base of the pack cementation method is the chemical reaction kinetics with increasing percentage of the halide activator in powder composition, and the reaction needs time and temperature for building the coating on the surface of the substrate [19]. At high treatment temperature, the thickness of the silicide layer increases and due to being at high temperature, halide activator is vaporized then reacting with the silicon powder [20]. The treatment time has direct effect on the coating thickness with increasing the treatment

*P* values less than 0.0500 indicate model terms are significant. In this case A, B, C, AB, AC, BC, A<sup>2</sup>, C<sup>2</sup> 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. Tables 3 and 4 show the analysis of the variance for the outputs.

A, B and C show the halide percentage, time and temperature respectively. To check the above mentioned reaction, ANOVA (analysis of variance) was used. Fig. 2a-b depicts the perturbation graph for the inputs and their effect.

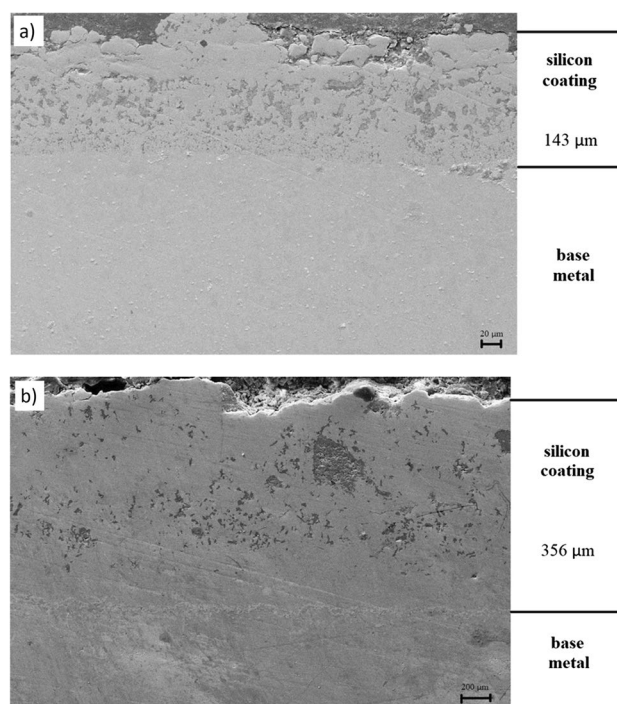
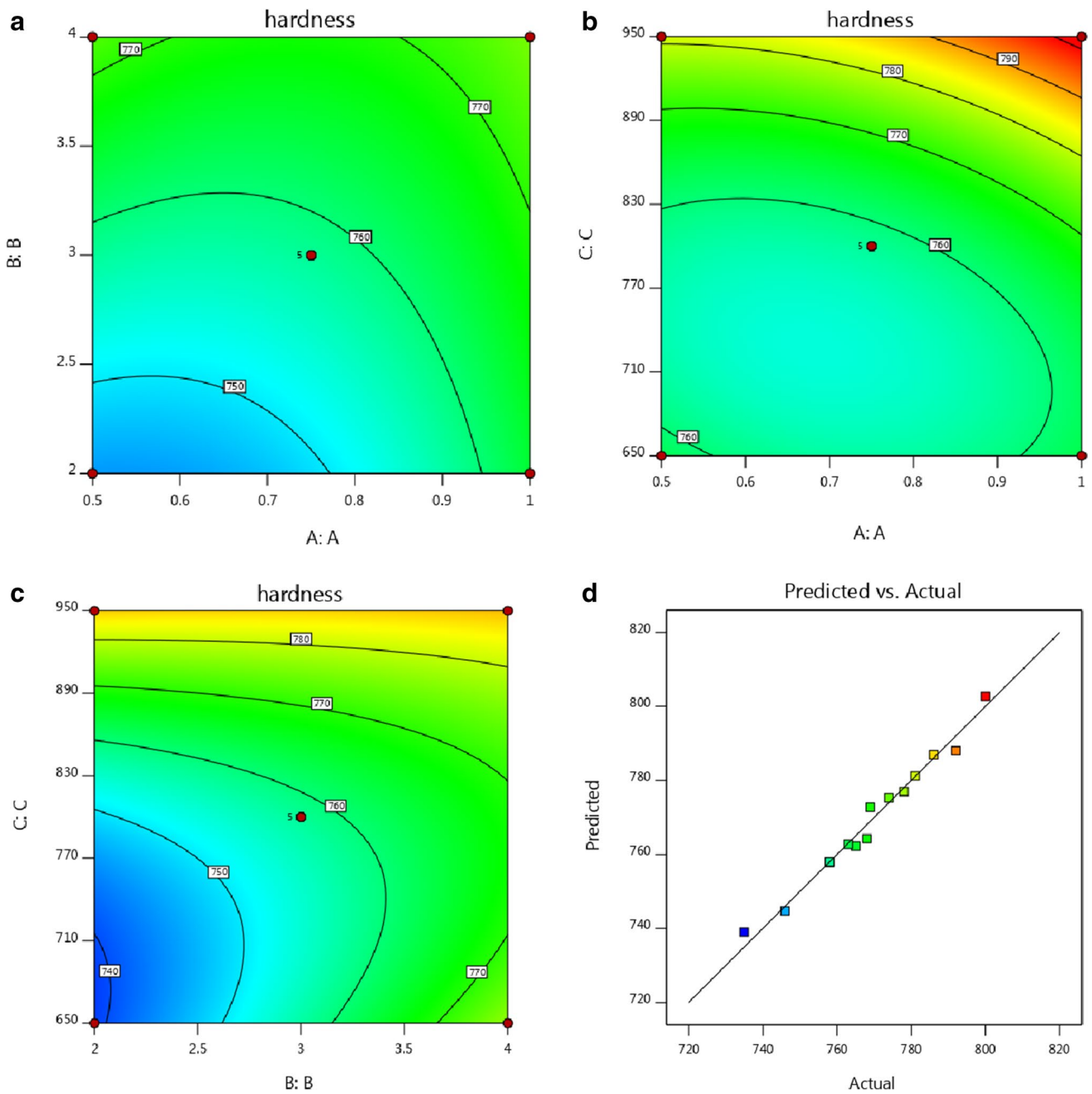


Fig. 4 Thickness distribution of silicon coating



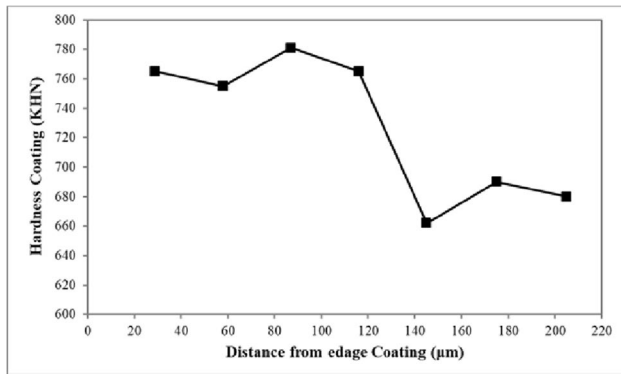


**Fig. 5** Interaction between time and halide (a), Interaction between temperature and halide (b) Interaction temperature and time (c), predicted and actual values

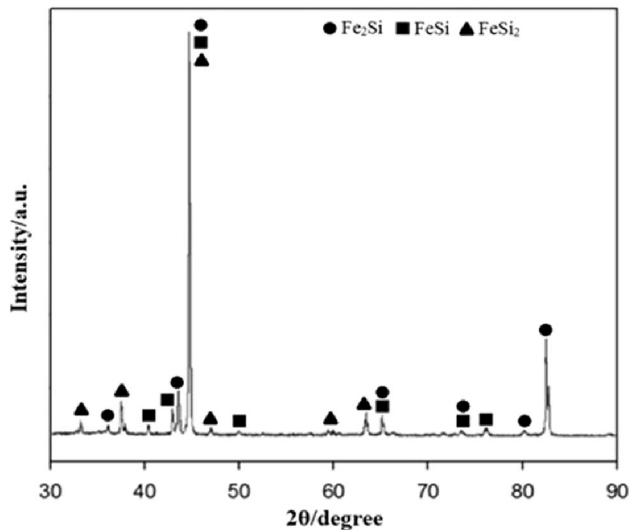
time; the active vapor has enough time to react with the surface of the substrate by building the coating on the surface [21]. Fig. 4a and b show the coating aspect for 0.5 and 1 wt.% of halide activator addition. The thickness of the coatings for the 1 wt.% of halide activator is higher than in the case of the addition of 0.5 wt.%.

### 5 Effect of Process Parameters on the Hardness

Figure 5 shows the effect of the process parameters on the hardness of the coating. Three specific parameters



**Fig. 6** Variation of micro-hardness after siliconizing



**Fig. 7** X-ray diffraction spectrum after siliconizing

have effect on the hardness of the coating: (i) treatment temperature, (ii) treatment time, and (iii) halide activator percentage used in the powder composition [17]. As the halide activator percentage increases, the silicon reactions kinetics improve. So, the active vapor is more effective as the treating time and temperature increase. Therefore, with increasing all the three specific parameters, the diffusion of the silicon improves [22]. The ordinary diffusion of the silicon supports the growth of the silicide layer inside the substrate.

The growing of the silicide layer improves the hardness of the substrate.

Figure 6 illustrates the profile of the micro-hardness from silicide layer to the steel substrate. The average micro-hardness of silicide layer (770 HV) is higher than the iron matrix one (677 HV). The specific reason for increasing hardness after siliconizing is the formation of Fe-Si intermetallics on the surface of the substrate. The silicon coating substantially increases the hardness [23, 24].

Figure 7 shows the XRD analysis of the silicide coating. The XRD detected three specific phases such as FeSi, Fe<sub>2</sub>Si and FeSi<sub>2</sub>. The formation of the phases indicated by the XRD pattern improves the hardness of the substrate surface [25].

Figure 8 shows the EDX profiles of the cross-section of the samples after siliconizing. The percentage of the silicon inside the surface of silicide layer shows the higher percentage of the silicon in the substrate. This clearly shows the penetration of silicon during the coating procedure.

To find the desirability of each response, eq. 3–5, were used to address the desirability of each output. This function has the ability to be changed due to weight of each input. For the optimization, input ranges are modified between the given ranges to find the best combination of parameters. So, to find the maximum and minimum values for the optimization, eq. 3 and 4–5 are used respectively.

When the target is maximum:

$$d_i = \begin{cases} 0 & Y_i < Low_i \\ \left( \frac{Y_i - Low_i}{High_i - Low_i} \right)^w & Low_i < Y_i < High_i \\ 1 & Y_i > High_i \end{cases} \quad (3)$$

When the target is minimum:

$$d_i = \begin{cases} 1 & Y_i < low \\ \left( \frac{Y_i - Low_i}{High_i - Low_i} \right)^w & Low_i < Y_i < High_i \\ 0 & Y_i > High_i \end{cases} \quad (4)$$

$$D = \left( \prod_{i=1}^n d_i^{r_i} \right)^{1/\sum r_i} \quad (5)$$

The “di”, “wi”, “D”, “r” show the unique desirability of each response, weight field, desirability objective function and importance. Table 5 elucidates the used data for the optimisation.

Having analyzed the results, the optimal combination of data is achieved based on Table 6. To check the accuracy



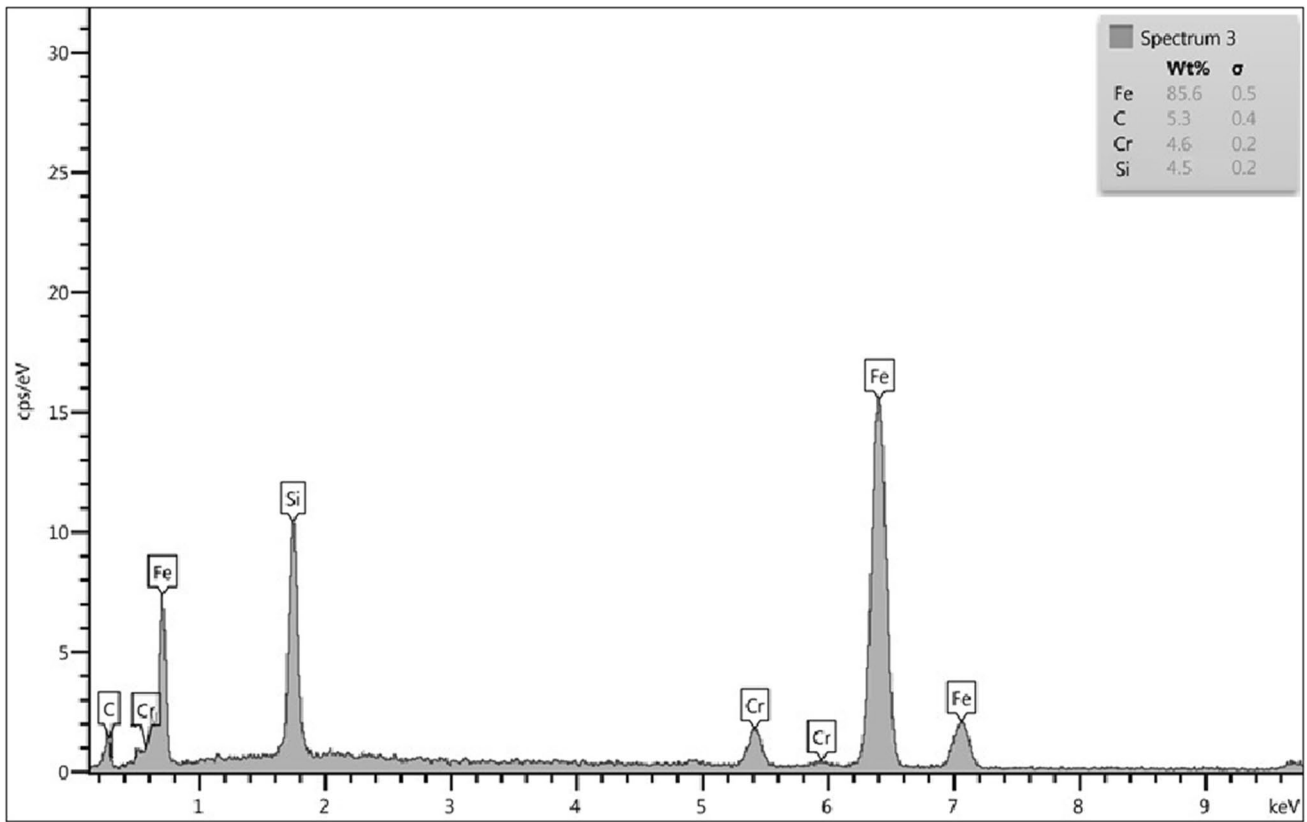


Fig. 8 EDS profile of the Cross-section after siliconizing

Table 5 Weight and importance of parameters

| Name                | Goal        | Lower Limit | Upper Limit | Lower Weight | Upper Weight | Importance |
|---------------------|-------------|-------------|-------------|--------------|--------------|------------|
| A: Halide activator | is in range | 0.5         | 1           | 1            | 1            | 3          |
| B:time              | is in range | 8           | 12          | 1            | 1            | 3          |
| C:temperature       | is in range | 650         | 950         | 1            | 1            | 3          |
| thickness           | maximize    | 12          | 356         | 1            | 1            | 5          |
| hardness            | maximize    | 680         | 800         | 1            | 1            | 5          |

Table 6 Desired parameters

|   | A: Halide activator | B:time | C:temperature | Thickness | Hardness | Desirability |
|---|---------------------|--------|---------------|-----------|----------|--------------|
| 1 | 1.000               | 3.575  | 950.000       | 317.957   | 800.883  | 0.941        |

of achieved data, a confirmatory test is done as shown is Table 7 and Fig. 9a and b.

As shown in the Table 6, there is a reasonable correlation between the achieved data and confirmatory test.

Table 7 Confirmatory tests

| Siliconizing performance | Units      | Desirability function | Experiment |
|--------------------------|------------|-----------------------|------------|
| Thickness                | Micrometer | <b>317.957</b>        | 325.34     |
| Hardness                 | GR         | <b>800.883</b>        | 812.67     |

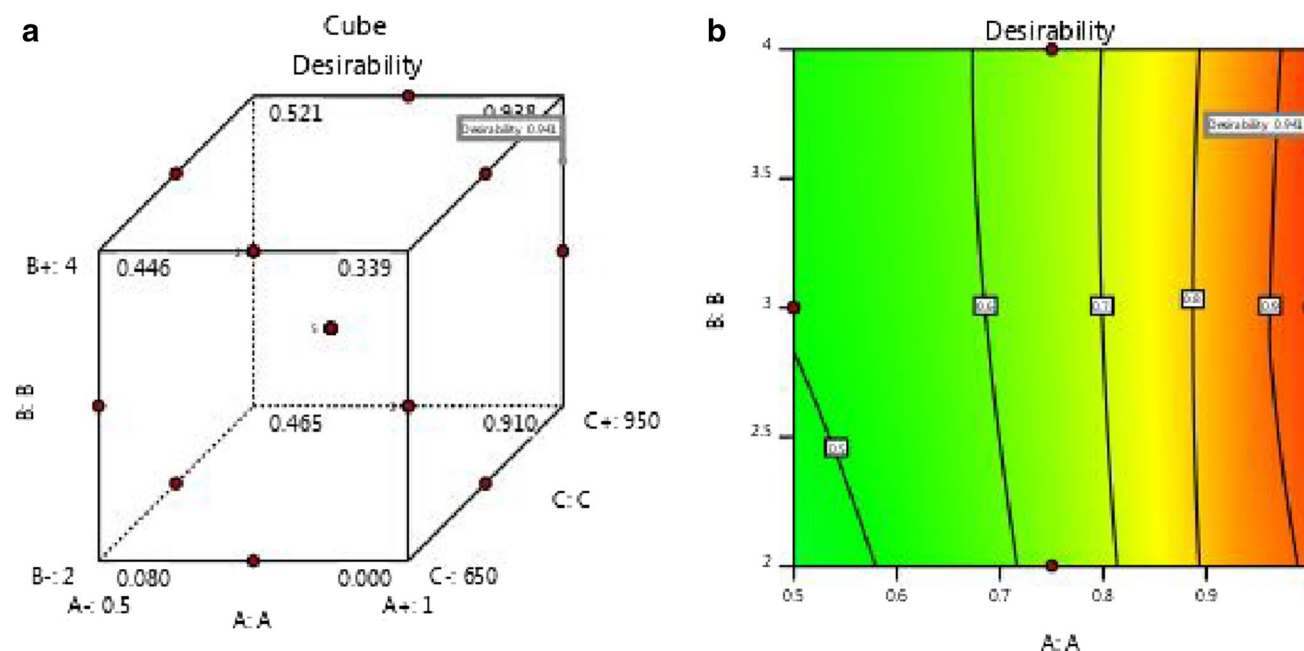


Fig. 9 Graph of desirability of the process

## 6 Conclusions

In this study, by using experimental tests and an optimization approach, the best combination of parameters for pack silicizing of AISI D2 tool steel was found. The silicide layer, fabricated by pack cementation method, was optimized in the employed process parameters, the percentage of the halide activator, treatment time and temperature at a fixed percentage of Si in the powder compound. The obtained results are summarized as follows:

1. With increasing the percentage of the halide activator, treatment time and temperature the thickness of the silicide layer increases slightly.
2. The Fe-Si intermetallics, fabricated by silicizing the surface, lead to a remarkable increase of the hardness of the surface samples.
3. By increasing the percentage of the halide activator inside the powder compound, treatment time and temperature the thickness of the sample increases and the thickness has a direct effect on the hardness profile.
4. The percentage of the silicon increased by increasing time, temperature and halide activator percentage.

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**Data Availability** Data available on request from the authors.

## Declarations

**Competing Interests** The authors have no relevant financial or non-financial interests to disclose.

**Ethics Approval** Not applicable.

**Consent to Participate** Not applicable.

**Consent for Publication** Not applicable.

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