Int J Adv Manuf Technol (2011) 56:397–409
DOI 10.1007/s00170-011-3167-z

ORIGINAL ARTICLE

Crack avoidance in steel piston rings through the optimization of process and gas nitriding parameters

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Received: 23 October 2009 / Accepted: 11 January 2011 / Published online: 29 January 2011

Abstract This paper describes research into adequately estimating the main variables of a thermochemical gas nitriding process of stainless steel parts for engine components. The paper lays out an experimental strategy for the nitriding process that optimizes a set of variables that have a bearing on the occurrence of nitriding cracks. The results demonstrate that several factors and interactions are relevant in the occurrence of nitriding cracks. The proposed strategy was found to be effective at achieving continuous improvement and stricter control.

Keywords Nitriding · Cracks · Piston rings · DOE

1 Introduction

Piston rings are metal pieces that, when installed in pistons inside engine cylinders, become circular and self-expansible. They provide a movable sealing between the combustion chamber and the carter of the engine. These components are submitted to intense functional demands. Ideally, piston rings should demonstrate low wear and keep structural and functional integrity under high temperatures and pressures. To help meet such demands, there is a technique that provides the contact face with the cylinder of these components with coatings or surface treatments to improve the wear and the corrosion resistance. Engineers of modern engines, in trying to lessen friction, tended to reduce the thickness of the piston rings, dropping even lower than 1.2 mm. This tendency led engineers to favor, for the compression rings closer to the combustion chamber, the use of steel—specifically, martensitic stainless steels. A special treatment has improved these rings' tribological properties; that treatment is called gas nitriding.

Gas nitriding transfers to steel intermediate superficial properties, properties shared by metallic and ceramic materials. Most important of these properties are low attrition coefficient and high resistance to both adhesive and abrasive wear. The process of producing these piston rings can be affected by several variables or factors. Each of which has either a direct or indirect impact on the quality of the end result. Given the high number of factors in these productive processes as well as their interactions, multiple disarrays are observed. Such disarrays lead to quality losses, rejection of pieces, and flaws in service, even in cases where nothing is detected internally with the productive system. A potentially catastrophic problem stemming from these disarrays are nitriding cracks in the piston rings. This work's objective is to devise an experimental strategy that yields an appropriate estimate of the main factors involved in gas nitriding, gas nitriding as it is applied to stainless steel piston rings. The developed

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strategy optimizes a group of variables that minimizes the occurrence of nitriding cracks.

2 Metallurgical aspects of nitriding and of cracks

In certain manufacturing processes—thermal treatment and machining, for example—established functional relationships exist between a set of input variables and output characteristics. Given the vast complexity of these relationships, manufacturing processes are eminently multivariate. In industrial settings, occasional instabilities in these complex processes can lead to significant quality loss and resource waste. A manufacturing process that has seen great advances in recent years is gas nitriding. Nitriding is a process of superficial diffusion. Treated workpieces are protected against wear, impact, and corrosion. Nitriding was developed early in the twentieth century, though precise methods of controlling its process were lacking at that time. It remained, until the 1980s, rather underutilized. Today, gas nitriding of engine parts, especially piston rings, has become a widely-used treatment. This is evident in Asia where a large number of patents have been filed, including, among others, US 4.557.492, US 5.013.371, and EP 0.588.558 B1.

Steel nitriding is a thermochemical process that diffuses nitrogen atoms into the crystal structure of a base metal. This diffusion hardens the metal by two means: it distorts the metal matrix and it precipitates compounds between the iron atoms (and/or alloy elements) and the nitrogen atoms. These compounds are called nitrides. Gas nitriding is fundamentally carried out in a furnace permeated with ammonia (NH_3). When ammonia comes in contact with heated metallic surfaces, say the retort of a furnace and the workpieces, it dissociates. This dissociation yields nascent nitrogen that diffuses into the metal and which partly migrates into the crystal structure. This diffusion, by partially forming iron nitrides and other alloy components, is what hardens the alloy. In most commercial processes, two factors controlled the degree of dissociation: the temperature and the flow of gas.

Since the early days, atmospheres were composed basically of ammonia. This offered little flexibility in terms of controlling the process. In some cases, excessively thick and brittle superficial layers were formed. In such layers, called white (or compound) layers, some porosity was occasionally observed in the workpieces, a fact which was still common even in the 1980s [1].

These white layers are formed through the contributions of four factors: (1) the temperature of treatment, (2) the composition of the internal atmosphere of the furnace (as a consequence of the activity of nitrogen), (3) the composition of the steel being treated, and (4) the duration of the treatment [2]. Beginning in the late 1960s, researchers tried to diminish the occurrence of white layers by developing a number of commercial purpose processes. These mixed ammonia with other gasses, such as nitrogen, carbon monoxide, hydrogen, and so on. By varying the amounts and combinations of these gasses, researchers mitigated the flaking and rupturing of the hard, fragile, and porous white layers [3]. Given, however, the crude control of the processes and atmospheres, such alternatives did little more than minimize the chances of obtaining the unwanted effects.

Figure 1 shows an example of a nitrided layer in a sample of martensitic stainless steel. The sample fundamentally contains 13% chromium and 0.6% carbon, alongside the white layer. Here the white layer is compact, well-bonded and thin, on average up to 4 μm thick.

New processing methods, developed over the past 30 years, have ushered in a resurgence of gas nitriding. A large number of patents have been issued [4], particularly in the area of ion (plasma) nitriding and controlled gas nitriding. Researchers in these fields, now possessing a deeper understanding of the fundamental mechanics of interaction between metal and atmosphere, have developed better control systems and methods [5].

One advantage to gas nitriding is that it causes insignificant distortion in the workpieces. This advantage is procured by the process's being carried out at temperatures generally lower than 600°C. Such temperatures, for steel, are relatively low and can eliminate the need for later processing. Further advantages include good wear reduction properties, better resistance to fatigue and corrosion, and a good appearance of the workpieces.

Key to the recent success of steel nitriding is the ability to effectively control (S. S. [6]), in the superficial layer of the treated workpieces, the concentration of active nitrogen. In some recent processes, control of the activity of nitrogen in the atmosphere has enabled control of the activity of

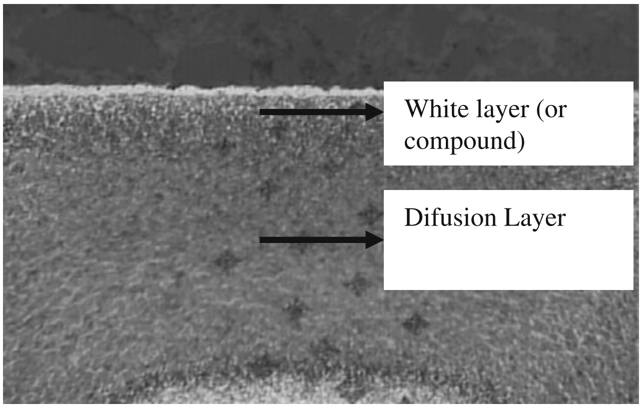


Fig. 1 Martensitic stainless steel nitrided cross-section nital 3% etched (×500). Source: Mahle Engine Components—Brazil

nascent nitrogen, a determining factor in obtaining the nitrided layer [1, 2, 7]. The technology that has helped introduce this control takes measurements during the process and adjusts a factor called nitriding potential (Np). Np is the ratio between the partial NH_3 and hydrogen pressures. Controlling the Np, which indicates the actual ammonia dissociation rate, allows predictable nitrided layers to form. The structure, depth, and hardness of these layers are replicable in tool steel. Tool steel is far richer in alloy elements such as tungsten, chromium, molybdenum, etc. Also rich in alloy elements is stainless steel, the focus of the present study.

Stainless steel's main element is chromium. The workpieces used here favor its properties of hardening, resistance, toughness, and corrosion resistance. Also, it is distorted only slightly by heat. Chromium, like carbon, is found in significant amounts in martensitic stainless steel. Together, the two elements produce the excellent qualities described above.

Resistance to adhesive and abrasive wear in stainless steel that contains chromium is significantly increased by gas nitriding. This is attributed to the formation of chromium nitrides with high degrees of hardness and to the superficial stress on the outer layer. Nitriding is often used as a case-hardening treatment so as to introduce residual compressive stresses into the outer surface of the workpiece. A gradient of stress, the so called tensile stress, occurs inside the substrate and core (Bekir Sami [8]). Tensile stresses occur when the nitrided layer expands and reacts with the non-nitrided zone. Chromium nitrides begin to precipitate after nitrogen atoms migrate, diffuse into, and saturate chromium-alloyed steels, such as martensitic stainless steels. The amount and gradient of that concentration of nitrogen and nitrides determine the level and profile of the residual stresses in the workpieces, as well as the hardness profiles. Contributing to the desirable occurrence of nitrogen diffusion and consequently nitrides is the proper control of the atmosphere and of the process [3, 5].

Nevertheless, for engine components, such control has proved to be highly complex, especially when it is applied to stainless steel. State-of-the-art techniques as well as equipment fail to prevent random instabilities that stem from a diversity of factors and their interactions. Sometimes, such instabilities give rise to unacceptable nitriding cracks in the workpieces. Nitriding cracks are sharp and deep, generally running the full thickness of the nitrided case from surface to core, usually more than 50 μm deep. Thus, entire lots or batches of components must be rejected. Figure 2 shows a roughly 50-μ deep-nitriding crack on a section of a nitrided piston ring, seen in an optical microscope without chemical etching. The piston ring cross-section shown is made of martensitic stainless steel, type AISI 440 B, containing approximately 0.9% carbon and 17% chromium. Figure 3 shows the cracked surface of the nitrided piston ring seen in a stereo microscope, ×20.

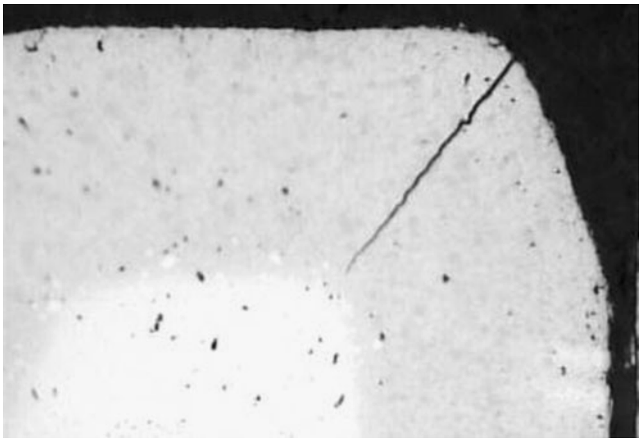


Fig. 2 Nitriding crack on nitrided stainless steel piston ring, without etch (×500)

3 Experiments and analysis

Given the large number of variables involved and the complexity of the potential interactions between them, the natural choice of methodology is design of experiments (DOE). Solutions to problems in industrial processes can be reached more easily and robustly when the experiments are planned and the results analyzed through statistical methods and techniques. They also point out a sequence of steps to be taken when conducting optimization work, concisely described as follows: (i) recognition and definition of the problem; selection of factors, levels and ranges; (ii) selection of the response variables; (iii) selection of the experimental design, experiment's execution, statistical analysis of the data; and (iv) conclusions and recommendations. The sequence is normally interactive and flexible. Some of the steps may be carried out simultaneously or in an inverted order without detriment to the expected results.

Considering the recognition and definition of the problem, the case in hand may be described as follows:

> Catastrophic failures, with severe consequences for both users and manufacturers of piston rings, may arise from the existence of long nitriding cracks in stainless steel engine component parts. By what variables in the gas nitriding process are such nitriding cracks caused? How can such variables be controlled?

The stainless steel wire from which piston rings are produced can present random surface defects—chips, bends, small cracks—that arise out of the wire rolling

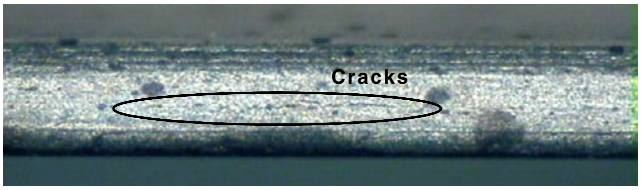


Fig. 3 Surface aspect of cracked part (×20)

process. These raw material defects are not rejected when smaller than 20 μm, in accordance with the wire suppliers and confirmed by fatigue tests.

3.1 Screening design

To curb the occurrence stamp out the emergence of these nitriding cracks in case-hardened stainless steel engine components, the team took into account a wide range of variables in the gas nitriding treatment and other operations in the piston rings process flow. Table 1 shows the list, evaluating the majority of potential variables likely to interfere with their occurrence.

The study's factors materialized from a brainstorming session with ten participants. In addition to the production engineers and operators involved in the everyday operation of the manufacturing process, the ten also included specialist technicians and metallurgists. The group met three times to discuss and better understand how the cracks occurred. As a result, all the potential factors which might influence the parts processing and the problem were gauged. Initially the team proposed the 43 factors seen above. They also defined and ascribed to each factor a *sensitivity* index associated with the controllability of the variables in the process (uncontrollable variables [0], medium-controllability variables [1], and controllable variables [2]).

Even though the factor "Metallurgical Analysis method" was rated 2*, there was broad consensus among team members that poor preparation would lead to preparation cracks in the nitrided piston rings samples. These abnormal cracks would appear from poor quality of the cutting, grinding, and polishing of the metallographic sample. The team, seeing that the factor acted not on the manufacturing process, but on the evaluation, ultimately fixed it.

After tabulating the variables, the team decided to experimentally investigate all factors rated 2; that is, all factors likely to be controlled. For these 18 factors, a resolution III Plackett–Burman fractional factorial design (using the minimum amount of 20 runs) or, with potentially

Table 1 Potential factors analysis and evaluation

Factor	Symbol	Sensitivity	Factor	Symbol	Sensitivity
Raw material cold work hardening		1	Cutting fluid at RTPF	*Cutt_Fluid*	2
Steel grade AISI 440 B×AISI 420	*AISI*	2	Cutting fluid at side grinding		1
Stresses from coiling		1	Inlet and exhaust of NH_3		1
Coiling diameter	*Coil_Diam*	2	Quality of NH_3		1
Wire width		1	NH_3 dissociator		0
Raw material residual stresses		0	Start of new furnace at line		1
Nitriding temperature	*Nit_Temp*	2	kN sensor (gas analyzer)		1
Nitriding atmosphere	*Nit_Atm*	2	NH_3 pressure	*NH_3_Press*	2
Temperature controller at furnace		1	NH_3 flow rate	*NH_3_Flow*	2
Room temperature		0	Nitriding tooling		1
Side grinding in-feed	*S_G_In_Feed*	2	Brushing pressure	*Brush_Press*	2
Side grinding wheel	*S_G_Wheel*	2	NH_3 pipeline (pipe resistance, etc..)		1
Metallurgical Analysis method		2*	Furnace pressure	*Furnace_Press*	2
RTPF—in-feed	*In_feed*	2	Temperature homogeneity		1
RTPF—grinding wheel	*Grind_Wheel*	2	Mass flow controllers		1
RTPF×lapping	*Lapping*	2	Stress relief tooling		1
Quantity of rings at furnace	*Quant_Rings*	2	Nitriding time		1
Stress relief temperature	*St_Re_Temp*	2	Parts cutting		1
Heating speed at furnace		1	Tooling racks assembling		1
Nitriding activation temperature (NGAS second stage)	*Nit_Ac_Temp*	2	Magnetic residue at rings	*Mag_Stat*	2
Nitrogen profile at rings		1	Gas burner triple X single		1
Parts area at furnace		1			

the same results, a L_{32} Taguchi design (with 32 runs) could be used.

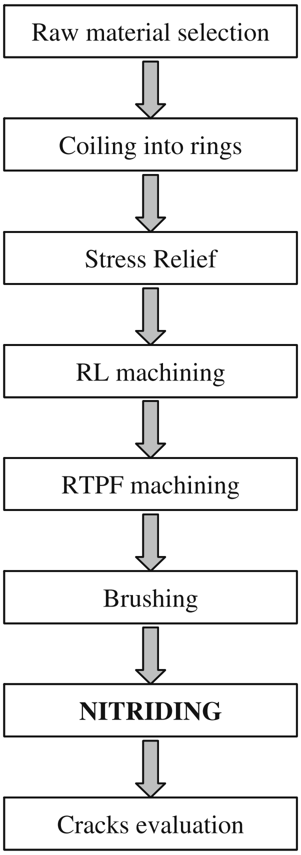
Figure 4 shows the processing sequence of the workpieces used in all experimental rounds. First, all workpieces used in all experiments were manufactured from a single spool of stainless steel wire. Next, so as to guarantee the uniform observance of parameters and minimize any effects beyond control, all the workpieces used in the tests were shape coiled, under a specialist's supervision, in a single sequence. According to the planned flow, the workpieces were heat-treated for stress relief, which is one of the studied factors here.

The Plackett–Burman design was then used and the experimental results are presented in Table 2 and Fig. 5.

From this screening analysis, some results were established:

1. The five factors (*RTPF–grinding wheel*, *steel grade AISI 440 B X AISI 420*, *side grinding wheel*, *RTPF x lapping*, *and coiling diameter*) were considered significant and their levels were established for future designs, as shown on the Pareto chart.
2. The six factors (*cutting fluid at RTPF*, *quantity of rings at furnace*, *furnace pressure NH_3 pressure*, *nitriding atmosphere*, *and NH_3 flow rate*) were not considered significant and further designs could either eliminate or define them as noise factors. These are the factors which effects were constant for the factor's level.
3. The seven remaining factors (*brushing pressure*, *nitriding activation temperature*, *RTPF—in-feed*, *stress relief temperature*, *side grinding in-feed*, *magnetic residue at rings*, *and nitriding temperature*) were considered borderline. It was insufficiently clear to either eliminate or select each factor's level. With these, further investigation is needed.

Fig. 4 Process flow



3.2 Fractional factorial design

The decision about further investigation is resumed in Table 3. It was reduced from seven to five the borderline factors judged to wield influence on the occurrence of nitriding cracks. The reduction amounted to three factors being combined (*brushing pressure*, *RTPF—in-feed*, *side grinding in-feed*). All three are connected with the machining of the piston rings' faces before the nitriding operation. Combining these three into one (named simply *in_feed_ rate*) was deemed much simpler than combining all factors, where all of them would be defined as either low level or high level. Table 3 is the result of this factor selection stage. It shows how the team decided for each factor—to study or to fix.

The fixed factors were held under check and control so as to have a minimal influence on the experiments. Table 4 shows the strategy to block the factor effects on further experiments.

Once the team had selected the variables, the specialists suggested two study levels for each of them. Their suggestions were grounded in their own experience with the manufacturing process, as well as in the shortcomings of the equipment and those found in the literature. Those levels were also chosen on the assumption that the nitrided layers should conform to the final customer's requirements of the parts. Table 4 displays the study factors and their corresponding levels selected for the pre-experimental stage. The variables are described briefly below.

- *Nitriding temperature*. Temperature maintained inside the furnace retort where the thermochemical treatment is carried out. In this stage of the process, the piston rings are subjected to a nitriding atmosphere for a defined period of time. During this time, the dissociation of ammonia with the consequent release of nitrogen atoms is expected to take place. As mentioned above, the nitriding temperature acts upon the degree of dissociation of the atmosphere as well as on the diffusion rate of nitrogen atoms into the base metal being treated.

Table 2 Plackett–Burman screening design for controllable factors

AISI	Coil_Diam	Nit_Temp	Nit_Atm	S_G_In_Feed (rpm)	S_G_Wheel	In_feed	G_Wheel	Lapping	Quant_Rings	St_Re_Temp	Nit_AcTemp	Cutt_Fluid	NH_3_Press	NH_3_Flow	Br_Press	Fur_Press	Mag_Stat	Nit_Crack
440	99	630	20%	0.010/2	Al 80 Mesh	0.01	Al 80 Mesh	Lapping	Full Batch	640	500	+10%Conc	+0.10 bar	+10%MasFl	2	1.1	De-Magn	121
440	100	550	20%	0.015/5	Al 80 Mesh	0.01	Al 80 Mesh	RTPF	50 parts	550	550	Regular	+0.10 bar	+10%MasFl	2	1.25	De-Magn	222
420	100	630	Regular	0.015/5	Al 220 Mesh	0.01	Al 80 Mesh	RTPF	Full Batch	640	500	+10%Conc	Regular	+10%MasFl	2	1.25	Magn	142
420	99	630	20%	0.010/2	Al 220 Mesh	0.1	Al 80 Mesh	RTPF	Full Batch	550	550	Regular	+0.10 bar	Regular	2	1.25	Magn	103
440	99	550	20%	0.015/5	Al 80 Mesh	0.1	Al 220 Mesh	RTPF	Full Batch	550	500	+10%Conc	Regular	+10%MasFl	1	1.25	Magn	135
440	100	550	Regular	0.015/5	Al 220 Mesh	0.01	Al 220 Mesh	Lapping	Full Batch	550	500	Regular	+0.10 bar	Regular	2	1.1	Magn	97
440	100	630	Regular	0.010/2	Al 220 Mesh	0.1	Al 80 Mesh	Lapping	50 parts	550	500	Regular	Regular	+10%MasFl	1	1.25	De-Magn	114
440	100	630	20%	0.010/2	Al 80 Mesh	0.1	Al 220 Mesh	RTPF	50 parts	640	500	Regular	Regular	Regular	2	1.1	Magn	182
420	100	630	20%	0.015/5	Al 80 Mesh	0.01	Al 220 Mesh	Lapping	Full Batch	640	550	Regular	Regular	Regular	1	1.25	De-Magn	92
440	99	630	20%	0.015/5	Al 220 Mesh	0.01	Al 80 Mesh	Lapping	50 parts	550	550	+10%Conc	Regular	Regular	1	1.1	Magn	88
420	100	550	20%	0.015/5	Al 220 Mesh	0.1	Al 80 Mesh	RTPF	50 parts	640	500	+10%Conc	+0.10 bar	Regular	1	1.1	De-Magn	133
440	99	630	Regular	0.015/5	Al 220 Mesh	0.1	Al 220 Mesh	RTPF	Full Batch	640	550	Regular	+0.10 bar	+10%MasFl	1	1.1	De-Magn	102
420	100	550	20%	0.010/2	Al 220 Mesh	0.1	Al 220 Mesh	Lapping	Full Batch	550	550	+10%Conc	Regular	+10%MasFl	2	1.1	De-Magn	61
420	99	630	Regular	0.015/5	Al 80 Mesh	0.1	Al 220 Mesh	Lapping	50 parts	550	500	+10%Conc	+0.10 bar	Regular	2	1.25	De-Magn	51
420	99	550	20%	0.010/2	Al 220 Mesh	0.01	Al 220 Mesh	Lapping	50 parts	640	500	Regular	+0.10 bar	+10%MasFl	1	1.25	Magn	6
420	99	550	Regular	0.015/5	Al 80 Mesh	0.1	Al 80 Mesh	Lapping	50 parts	640	550	Regular	Regular	+10%MasFl	2	1.1	Magn	127
440	99	550	Regular	0.010/2	Al 220 Mesh	0.01	Al 220 Mesh	RTPF	50 parts	640	550	+10%Conc	Regular	Regular	2	1.25	De-Magn	100
440	100	550	Regular	0.010/2	Al 80 Mesh	0.1	Al 80 Mesh	Lapping	Full Batch	640	550	+10%Conc	+0.10 bar	Regular	1	1.25	Magn	190
420	100	630	Regular	0.010/2	Al 80 Mesh	0.01	Al 220 Mesh	RTPF	50 parts	550	550	+10%Conc	+0.10 bar	+10%MasFl	1	1.1	Magn	115
420	99	550	Regular	0.010/2	Al 80 Mesh	0.01	Al 80 Mesh	RTPF	Full Batch	550	500	Regular	Regular	Regular	1	1.1	De-Magn	114

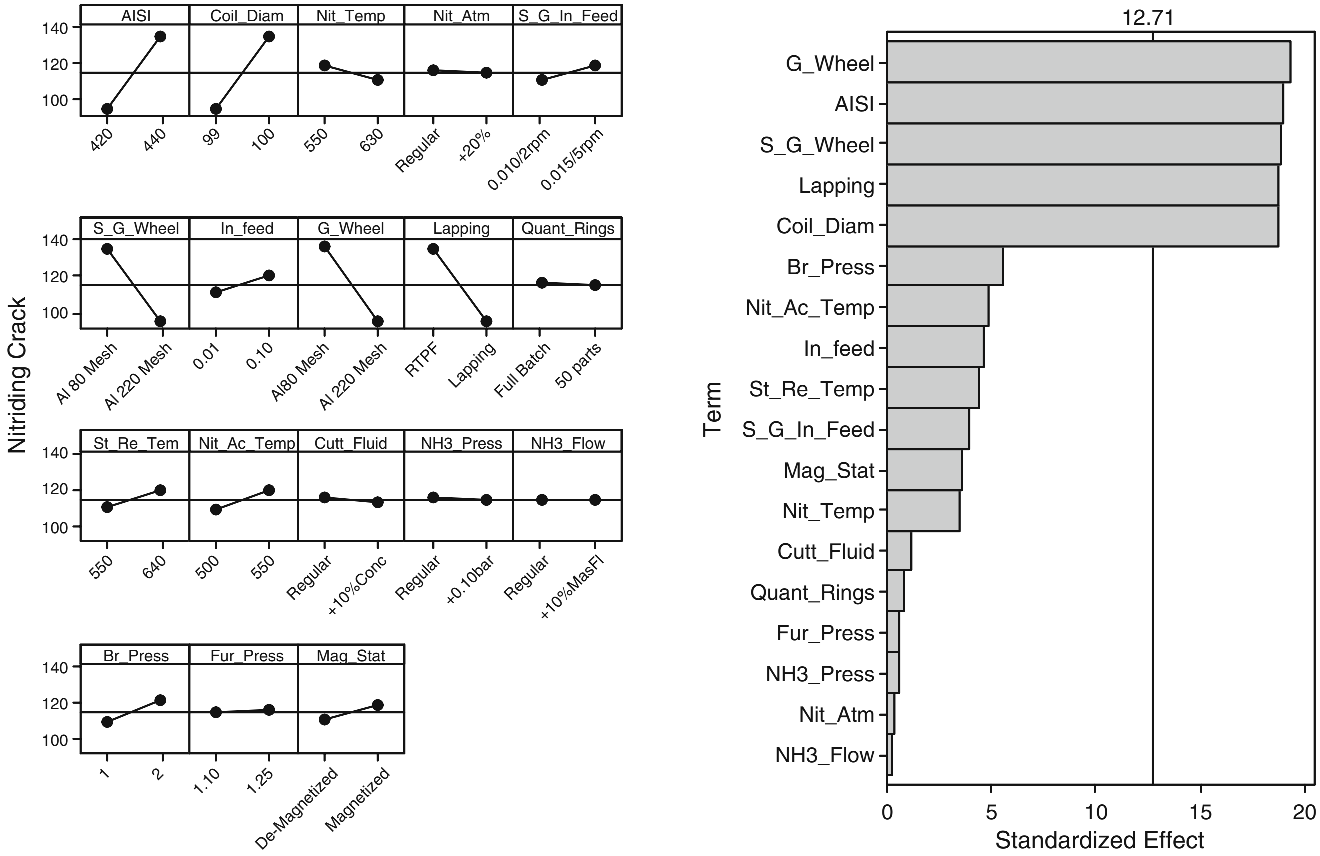


Fig. 5 Plackett–Burman screening results (main effects and Pareto chart)

Table 3 Factors selection for DOE after screening design

Factor	Decision	Level
Steel grade AISI 440 B × AISI 420	Kept	AISI 440 B
Coiling diameter	Study	Regular production
Nitriding temperature	Study	DOE
Nitriding atmosphere	Kept	Regular production
Side grinding in-feed	Study	DOE (Combined)
Side grinding wheel	Kept	Regular production
RTPF—in-feed	Study	DOE (Combined)
RTPF—grinding wheel	Kept	Regular production
RTPF × Lapping	Kept	RTPF
Quantity of rings at furnace	Kept	See text
Stress relief temperature	Study	DOE
Nitriding activation temperature (NGAS second stage)	Study	DOE
Cutting fluid at RTPF	Kept	Regular production
NH_3 pressure	Kept	Regular production
NH_3 flow rate	Kept	Regular production
Brushing pressure	Study	DOE (Combined)
Furnace pressure	Kept	Regular production
Magnetic residue at rings	Study	DOE

Table 4 Factors kept under control

Factor	Blocking control
Stress relief furnace	Selected the furnace number #1
Side gringing machining	Selected just 01 machine type RL, just one grinding wheel and same operator for all tests
Machining RTPF	Selected just one machine type RTPF, selected one tooling set, just one grinding wheel and same operator for all tests
Raw material wire	Same wire spool from one single batch of steel grade type AISI 440B, based on 17–18% Cr, 0.8–0.95% C, Mn and Si 1.0% max. And Mo 1.5% maximum. Wire as received quenched and tempered for 38–42 HRC
Brushing	Brush type same for all tests, selected one tooling set, just one machine and same operator for all tests
Metallurgical analysis method	Sampling, method and metallurgist kept the same
Nitriding	Selected the furnace number #2

- *In-feed rates on RL/RTPF/Brushing*. These machining stages are respectively RL, side grinding of the piston rings lateral faces with an abrasive grinding wheel; RTPF, profile grinding of the external faces named outer diameter; brushing to reduces roughness using plastic-bristled brushes containing abrasives. These stages, through the cutting strain or the polishing, may introduce residual stresses into the machined workpieces' base metal. Low in-feed rates subject the workpieces to lighter strain. More severe in-feed rates, here named high, may cause residual stresses, plastic deformations, etc. To relieve residual stresses, workpieces with high levels may develop nucleated cracks while undergoing work or hardening heat-treatments such as nitriding.
- *Magnetic status of the piston rings* (*workpieces*). When subjected to the strains inherent to machining, steel workpieces occasionally become magnetized. The tests included workpieces that were both magnetized (residually) and demagnetized (by electromagnetic coils). Looking at empirical evidence, team members suspected that the magnetized workpieces, when put through heat treatment with the diffusion of nitrogen atoms, might behave differently.
- *Stress-relief temperature*. Temperature to which the workpieces are exposed—in this study, for 90 min—to the heat treatment in a furnace with air or an inert atmosphere, seeking the relief of residual stresses resulting from the mechanical shape coiling of the steel wire. This temperature is set below the temperature range for tempering the raw material, so as not to allow any perceptible loss of hardness. Such a temperature for the steel used here is below 640°C. According to metallurgical practice and literature, the higher the stress–relief temperature, the greater the relief of residual stresses.
- *Nitriding activation temperature* (*NGAS second stage*). Lower than the full nitriding temperature, at this temperature gaseous ammonia is introduced to allow for early dissociation reactions on the surface of the workpieces and, consequently, the first nitrides nuclei and white layer [2]. In this step, the duration of which is clearly defined, the surfaces of the workpieces are activated as a means of permitting the absorption and diffusion of nitrogen atoms [1].

Closing the pre-experimental stage, the lengths of the nitriding cracks, observable through metallographic study and measured in microns, were defined as the response variable.

In selecting the experimental matrix, some options seemed natural choices. A complete factorial arrangement with $k=32$ rounds ($k=2^5$) would allow all main variables and their possible interactions to be analyzed without any aliasing between the effects of the proposed model. Such aliasing consists of the dubious interpretation of an effect or interaction with other interactions. In the occurrence of aliasing, one may take a particular effect in a response to be putatively caused by a variation of an input factor, when in fact the effect has been caused by an interaction. When it is wanted to decrease the number of experimental rounds, the

Table 5 Factors and levels for the second DOE

Factor or study variable	Symbol	Level (−)	Level (+)
Nitriding temperature	Nit_Temp	570 C	610 C
Stress relief temperature	St_Re_Temp	585 C	625 C
Nitriding activation temperature (NGAS second stage)	Nit_Ac_Temp	470 C	530 C
Side grinding in-feed/RTPF—in-feed/brushing pressure	In_feed_rate	Low	High
Magnetic residue at rings	Mag_Stat	De-magnetized	Magnetized

Table 6 Experiments and responses matrix

Run	Nit_Temp (C)	St_Re_Temp (C)	Nit_Ac_Temp (C)	In_feed_rate	Mag_Stat	R1	R2	R3	Average	SD
1	570	585	470	Low	Not	43	24	5	24.00	19.00
2	570	585	470	High	Yes	55	48	41	48.00	7.00
3	570	585	530	High	Not	195	232	213	213.33	18.50
4	570	585	530	Low	Yes	163	176	170	169.66	6.50
5	570	625	470	High	Not	59	59	59	59.00	0.00
6	570	625	470	Low	Yes	86	64	51	67.00	17.69
7	570	625	530	Low	Not	192	183	187	187.33	4.50
8	570	625	530	High	Yes	206	215	210	210.33	4.50
9	610	585	470	High	Not	5	48	0	17.66	26.38
10	610	585	470	Low	Yes	39	20	1	20.00	19.00
11	610	585	530	Low	Not	54	41	58	51.00	8.88
12	610	585	530	High	Yes	86	46	90	74.00	24.33
13	610	625	470	Low	Not	7	4	0	3.66	3.51
14	610	625	470	High	Yes	1	2	2	1.66	0.57
15	610	625	530	High	Not	66	76	82	74.66	8.08
16	610	625	530	Low	Yes	42	46	70	52.66	15.14

selection of a fractioned factorial is the second best option, despite the occurrence of aliasing. In this study, the fractioned factorial arrangement, with $k=2^{5-1}=16$ rounds has the *resolution order V*, where the main factors are aliased with fourth-order interactions. Given that the effects of fourth order interactions are negligible, such a resolution level for most experimental projects can generally be considered sufficient. That experimental project was therefore adopted in this research work.

Experiments with less resolution, as Plackett–Burman's (with $k=12$ rounds), or higher fractioned factorials with $k= 2^{5-2}= 8$ rounds are classified as resolution level III. At that level, the effects of the main factors are aliased with the effects of second order interactions, which cannot be overlooked. Another class of experimental planning that has restrictions in the presence of interactions but captures important robustness aspects is the Taguchi method. For comparison effects, this strategy has been adapted here.

Apart from the choice of experimental project, the replications of all rounds were deemed fundamental for analyzing the results. In the statistical analysis of the results, the existence of replications allows the use of hypothesis testing. Thus, as shown in Table 5, 16 experiments were conducted in three replications. The sequence of assays on the table shows a well-defined pattern. Yet being a cornerstone of design of experiments, a random sequence was actually used.

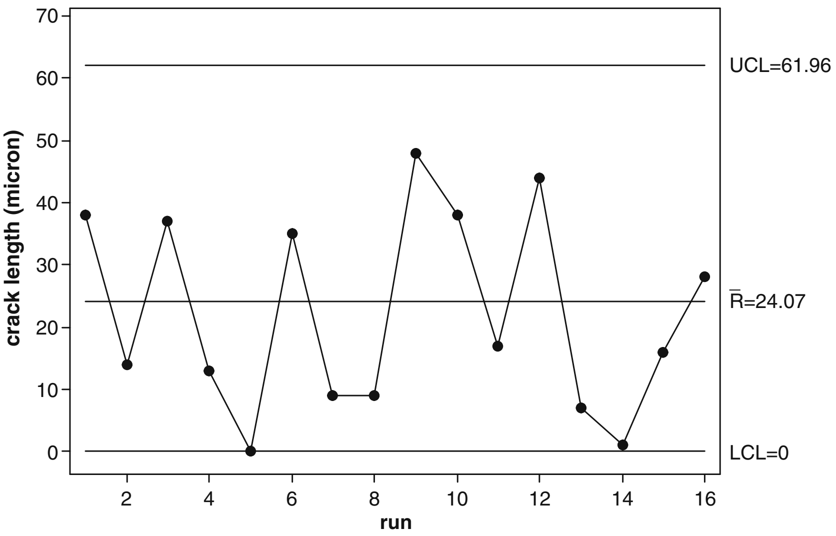


Fig. 6 *R* control chart

Table 7 Coefficient estimation and ANOVA for cracks size

Term	Efect	Coefficient	SE Coefficient	*T*	*P*	Significance (5%)
Constant		79.63	2.98	26.72	0.00	**
Nit_Temp	−85.5	−42.75	2.98	−14.34	0.00	**
St_Re_Temp	4.83	2.42	1.00	0.81	0.427	
Nit_Ac_Temp	99.08	49.54	2.98	16.62	0.00	**
In_feed_rate	15.46	7.73	2.98	2.59	0.017	**
Mag_Stat	1.54	0.77	2.98	0.26	0.798	
Nit_Temp×St_Re_Temp	−12.42	−6.21	2.98	−2.08	0.05	**
Nit_Temp×Nit_Ac_Temp	−46.67	−23.33	2.98	−7.83	0.00	**
Nit_Temp×In_feed_rate	−5.29	−2.65	2.98	−0.89	0.385	
Nit_Temp×Mag_Stat	−1.21	−0.6	2.98	−0.2	0.841	
St_Re_Temp×Nit_Ac_Temp	−0.5	−0.25	2.98	−0.08	0.934	
St_Re_Temp×In_feed_rate	−6.71	−3.35	2.98	−1.13	0.273	
St_Re_Temp×Mag_Stat	0.21	0.1	2.98	0.03	0.972	
Nit_Ac_Temp×In_feed_rate	12.54	6.27	2.98	2.1	0.048	**
Nit_Ac_Temp×Mag_Stat	−6.54	−3.27	2.98	−1.1	0.285	
In_feed_rate×Mag_Stat	−9.25	−4.63	2.98	−1.55	0.030	
$S=17.4124\times R^2(\text{adj})=93.65\%$						
Source	GL	Seq SS	Aj SS	Aj MS	F	P
Main effects	5	142,519	145,224	29,044.7	95.8	0.00
Second order interaction	10	22,944	22,944	2,294.4	7.57	0.00
Residual error	21	6,367	6,367	303.2		
Pure error	21	6,367	6,367	303.2		
Total	36	171,831				

The processing sequence of the workpieces was equal to the Plackett–Burman design. As seen in Table 6, the treatment that the workpieces underwent was divided into four blocks: two at 585°C and two at 625°C. The same furnace was used and, at all times, an operator monitored the parameters. Three replications of the four blocks were produced.

After all stress relief heat treatment rounds, the rings had their faces machined in the operations RTPF, Brushing and RL, according to the plan of experiments 1–16 in Table 6.

The figures seen in responses R1, R2, and R3 stand for the sums (in microns) of the crack lengths measured in 27 metallographic cross-sessions on workpieces systematically collected from each assay, from different same points of the nitriding furnace. All of the metallurgical analyses were performed by the same analyst using a single method of preparation and analysis.

Proceeding to the statistical analysis of the data, a variety of data and graphs may be used. The Minitab statistical software was mostly used here. A range control chart (R) shows the subgroup variation in the three replications, as seen in Fig. 6. Although chart R was under control, great oscillation within a subgroup is noticeable. That underscores the high degree of volatility of the data and the importance of controlling the variability of the process, represented by the standard deviation.

In Table 7 are seen the analysis of variance table and the estimate of the factors' effects on the length of nitriding cracks. This table summarizes the main effects and the second-order interactions responsible for a good polynomial model with an adjusted coefficient of determination of

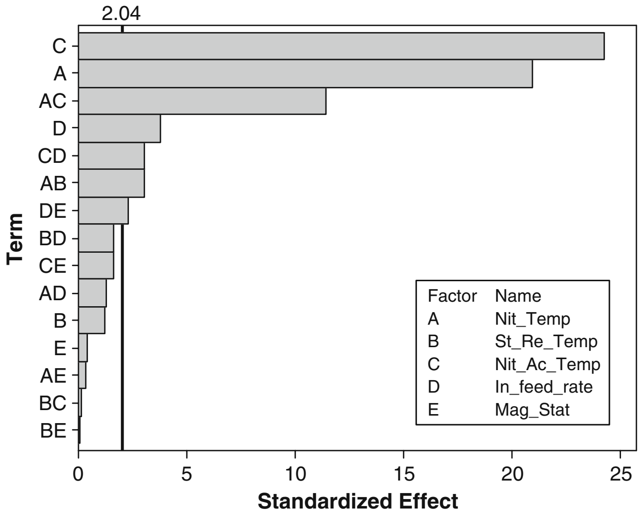


Fig. 7 Pareto chart for regression terms

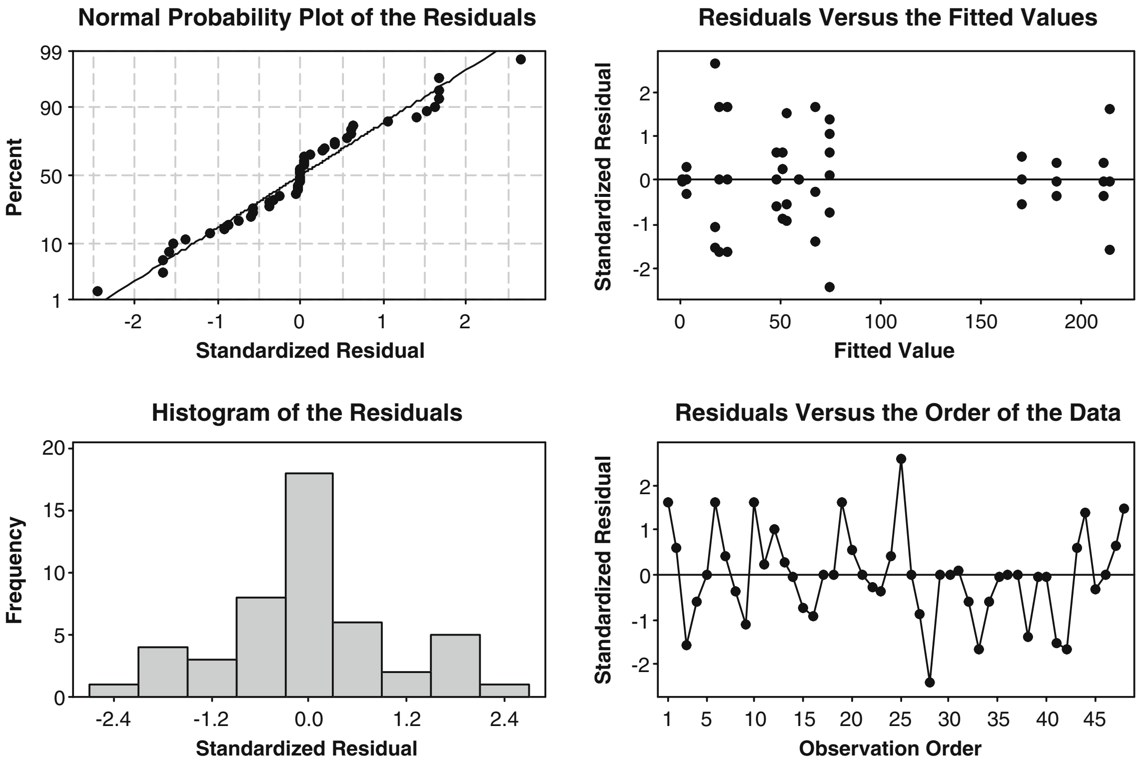


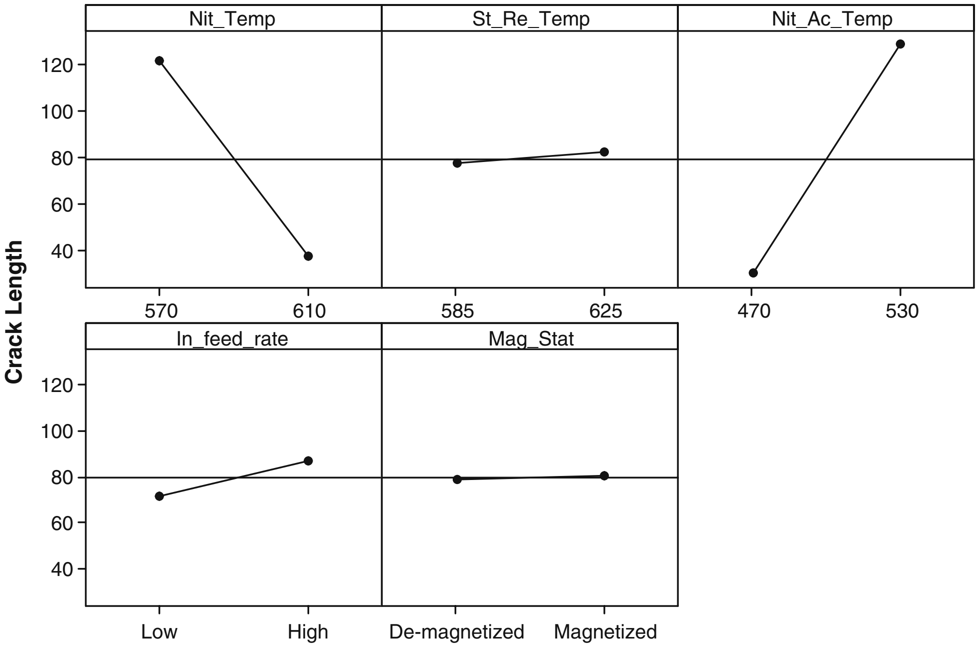
Fig. 8 Residual plots

93.65%. The *P* values are associated with tests of hypotheses, which reject, at a level of 5%, the null hypothesis of equality of the model's terms when smaller than the significance level. In this case, all factors in bold are considered significant to the occurrence of nitriding cracks.

The Pareto chart, in Fig. 7, represents the effects of the model's terms. The cut line (called length line) shows the significant effects considered in terms of the Student *t* figures.

The boldfaced factors and interactions in Table 7, also represented in the Pareto chart of Fig. 7, constitute the

Fig. 9 Crack occurrence average effects from factors



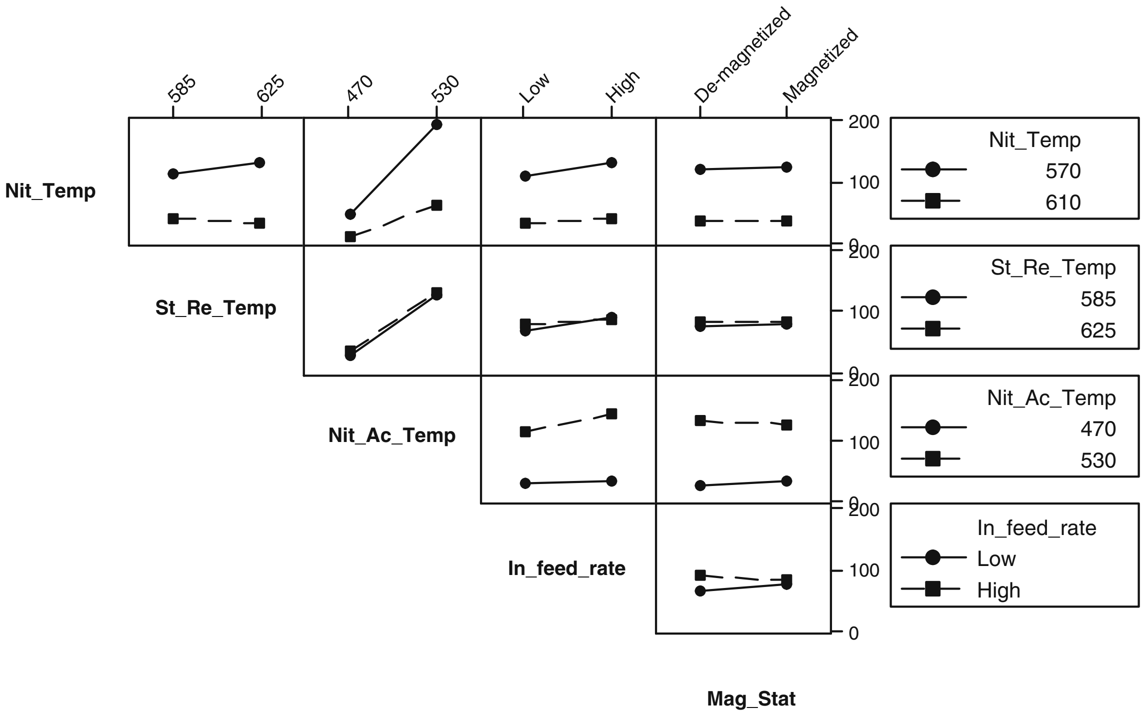


Fig. 10 Two-factor interaction plot

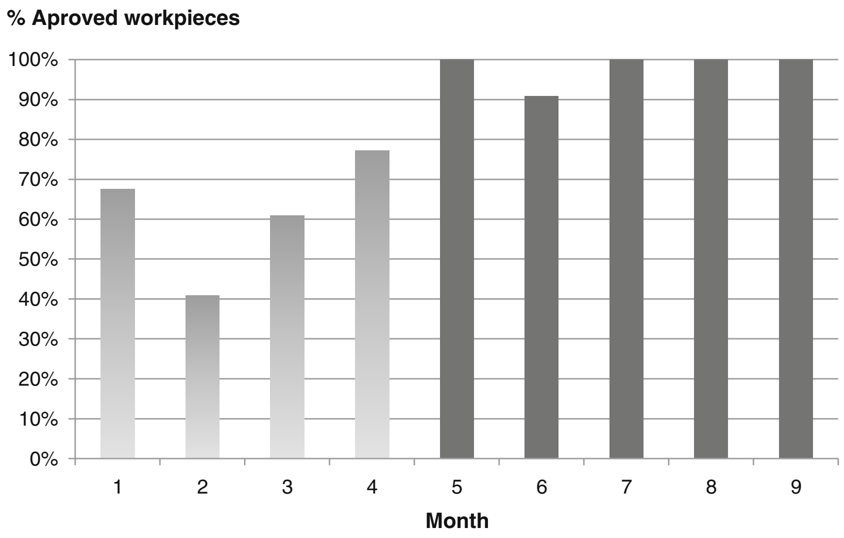
following regression model, which can represent the proposed problem. For the effects of forecasting, the figures are to be replaced with their coded variables (−1 and +1). These correspond to the levels described in Table 5. The analysis of residuals, fundamental to the definition of any regression analysis model, reveals that the residuals can be considered independent and normally distributed, as seen in Fig. 8.

$$\text{Nitriding crack length} = 79.63 - 42.75A + 49.54C + 7.73D - 6.21AB - 23.33AC + 6.27CD + 4.63DE$$

Figure 9, displaying results from after changing the levels of each one of the studied variables, clearly shows the changes' effect on the average occurrence of cracks. What stand out are the effects of two factors, *nitriding temperature* and *nitriding activation temperature* (*NGAS second stage*).

Second-order interactions can be observed in Fig. 10. In this graph, the non-parallel lines indicate the existence of interactions. Here, the most dramatic interaction is the one between the variables nitriding temperature and nitriding activation temperature (NGAS second stage).

Fig. 11 Approved rate after process upgraded



The best-known optimization method for multiple responses (Y_k) is the so-called Harrington's desirability function improved by Derringer and Suich [9]. In this method, the statistical model is first obtained using ordinary least square. Then using a set of transformations based on the limits imposed on the responses, a conversion is conducted for each one of the responses. This results in an individual desirability function d_i, with $0 \leq d_i \leq 1$. These individual values are then combined using a geometrical average, such as $D = (d_1(Y_1).d_2(Y_2).....d_k(Y_k))^{\frac{1}{k}}$. This value of D gives a solution of commitment and is restricted to the interval [0, 1]. D is close to 1 when the responses are close to its specification. The type of transformation depends on the desired optimization direction. The desirability function approach to a problem of optimization is simple, easy to apply, and allows the user to judge the importance of each response.

The optimized solution to minimize the length of cracks can be attained by using *desirability* function. The forecast *compound desirability* was 0.98 (where the figure 1.00 represents a perfect optimal point) and the global solution to the proposed problem, considering the last five factors to be defined, can be expressed as:

Nitriding temperature=610°C
Stress relief temperature=625°C
Nitriding activation temperature (NGAS second stage)=470°C
In-feed rates on RL/RTPF/brushing=Low
Magnetic status=De-magnetized

It is important to mention that the proposed solution take into account the best levels from all the other factors, defined from previous screening DOEs.

4 Conclusions

DOE has been found to be effective at achieving continuous improvement and stricter control when applied to metallurgical, thermochemical, and welding processes, processes that generally involve multiple acting factors. Several papers and publications attest to this claim. Here, the methodology has proved adequate. An initial exploratory analysis listed 43 factors that might, either in isolation or through interactions, influence the occurrence of nitriding cracks. Narrowing that number down to 5, the experimental investigation led to a fairly robust stage. After the conclusion of the experimental investigation, the levels of the study factors were adjusted to the optimal settings obtained through the use of Deringer's desirability function. The results were monitored in the following months. In Fig. 11, it can be seen a reflection of greater stability in the process level obtained in the study: during months 7, 8, and 9, no workpieces were rejected. The results demonstrate that several factors and interactions are relevant in the occurrence of nitriding cracks. What proved to be most effective in avoiding nitriding cracks, consistent with the literature, was adjusting to higher settings the factors nitriding temperature and stress relief temperature. On the other hand, one factor scarcely explored in specialized literature showed great influence on the results—the nitriding activation temperature (NGAS second stage). As a goal of future research work, a better understanding of this factor is suggested here.

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