Investigation of the Parameters Influencing the Refractoriness under Load (RuL) Testing Results for Refractory Materials

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This paper focuses on RuL (Refractoriness under Load) method, and the associated standards which are EN ISO 1893 for shaped materials and EN ISO 1927-6/9 for unshaped materials. The present investigation in RuL testing consists in performing a 2-steps campaign, the first step aiming at determining the relevant factors influencing the data (signal and noise) and the second one aiming at improving the signal values as well as the dispersion of data. The first campaign is performed by one laboratory which carries out an experimental factorial design plan with a comprehensive number of factors. The second campaign involves four different laboratories within a Round Robin Test via a design plan with a reduced number of factors.

1 Introduction

Up to now, the elaboration and the characterization of refractory materials are defined by a large panel of standards (EN, ISO …), which ensures as much as possible that different laboratories may obtain the nearest values of given properties for the same material, providing a strict use of the testing standards. Unfortunately, it can sometimes happen that the results provided by different laboratories present a quite significant deviation. In certain cases, there are substantial economic issues that may result in legal proceedings involving users, building-contractors and refractory producers. It is in this perspective of improvement of precision data that a European consortium of testing laboratories is created. The project ReStaR [1] includes a complete investigation of existing testing methods, in order to suggest upgrading of related EN standards in focusing on quality improvement, cost reduction, convenience in use and time saving.

2 Determination of influencing factors

The RuL EN standard specifies a method for determining the deformation of dense and insulating shaped/unshaped refractory products, when subjected to a constant load under conditions of progressively rising temperature, by a differential method. A cylindrical test piece is subjected to a specified constant compressive load and heated at a specified rate of temperature increase until a prescribed deformation or subsidence occurs. The cylinder is provided with a 12 mm-hole extending throughout the height, in order to measure temperature (by a thermocouple) at the geometric centre of the test-piece. The deformation of the test piece is recorded as the temperature increases, and the temperatures corresponding to specified proportional degrees of deformation are determined. The test may be carried out up to a maximum temperature of 1700 °C.

In considering the equipment, the test-piece preparation and the process conditions, the testing parameters (factors), which reasonably may have a potential impact on RuL properties are:

- Location of taking of test-pieces from the initial brick (extraction)
- Pressing/casting direction (shaping direction)
- Height of the sample (height)
- External diameter of the sample (diameter)
- Roughness of upper and lower sections of the test-piece (grinding)
- Loading stress applied on the test-piece during the process (loading)
- Position of the central thermocouple required to measure the temperature "inside" the test-piece and to identify the different RuL temperatures (thermocouple position)
- Shape of cast specimen from which the sample was extracted (shape).

Among the different existing forms of refractory materials, the authors have chosen to investigate:

- A dense brick from RHI = HA75 (B brick, High Alumina >75 % Al_2O_3),
- An insulating brick from RATH = LWI35 (Light Weight Insulating >35 % Al_2O_3),
- A dense castable from Calderys = MCC75 (Medium Cement Castable >75 % Al_2O_3), in pre-fired (1200 °C) or unfired state.

For the investigation of the signal effect of RuL testing parameters, it was decided to use the same design plan for the study of

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Tab. 1 Chart of investigated parameters and associated levels

N°	Factor	Level −1	Level +1
X1	Extraction	Corner	Middle
X2	Shaping direction	// testing direction	testing direction
X3	Height	30 mm	50 mm
X4	Diameter	40 mm	50 mm
X5	Grinding	Yes	No
X6	Loading	Nominal −10 %	Nominal +10 %
X7	TC	Center −20 mm	Center +20 mm
X8	Shape	A (230 mm × 114 mm × 64 mm)	B (230 mm × 55 mm × 64 mm)

Thermocouple position: TC

Tab. 2 Experimental matrix for shaped materials

exp	X0	X1	X2	X3	X4	X5	X6	X7
1	+	+	+	+	−	+	−	−
2	+	−	+	+	+	−	+	−
3	+	−	−	+	+	+	−	+
4	+	+	−	−	+	+	+	−
5	+	−	+	−	−	+	+	+
6	+	+	−	+	−	−	+	+
7	+	+	+	−	+	−	−	+
8	+	−	−	−	−	−	−	−

(+) corresponds to the upper level of the factor
(–) corresponds to the low level of the factor

Tab. 3 Experimental matrix for unshaped materials

exp	X0	X1	X2	X3	X4	X5	X6	X7	X8
1	+	+	+	−	+	+	+	−	−
2	+	−	+	+	−	+	+	+	−
3	+	+	−	+	+	−	+	+	+
4	+	−	+	−	+	+	−	+	+
5	+	−	−	+	−	+	+	−	+
6	+	−	−	−	+	−	+	+	−
7	+	+	−	−	−	+	−	+	+
8	+	+	+	−	−	−	+	−	+
9	+	+	+	+	−	−	−	+	−
10	+	−	+	+	+	−	−	−	+
11	+	+	−	+	+	+	−	−	−
12	+	−	−	−	−	−	−	−	−

(+) corresponds to the upper level of the factor
(–) corresponds to the low level of the factor

both shaped materials (dense shaped and insulating shaped). Concerning the unshaped material, one additional parameter was introduced – shape of initially cast specimen from which the samples were cut – so that a different design plan was implemented.

2.1 Choice of the design plan

The Tab. 1 presents the 2 levels associated to each of the 8 investigated parameters. The first 7 parameters will concern shaped materials, while all the 8 parameters (including shape of the initial brick) will concern unshaped materials.

The choice of a Plackett & Burman [2–3] experimental design for ReStaR phase 1 is well suited as it allows to determine the most relevant factors in a limited number of experiments with an easy way to calculate the main factors. The strict use of such a model does not allow any interactions between the factors – they are considered negligible – so only the main effects of the factors are estimated. Nowadays current computing resources may enable a relevant estimation of the interactions between the factors. The experimental matrix is based on a Hadamard's matrix. Its main interest is that each of the 2 levels of any factor has the same probability of occurrence – appears the same number of times – throughout all the experimental runs. Below are presented the experimental matrixes, which require respectively 8 runs or 12 runs, taking in account respectively the 7 factors (shaped) or 8 factors (unshaped).

For every combination of factors, and for all types of materials, a repeatability test was realised by the mean of 2 test-pieces per experiment. For example, 8 runs × 2 test-pieces i.e. 16 experiments were carried out for shaped materials.

3 Results

The different results are obtained by identification of the points at which the deformation measured from the highest point on experimental curve corresponds to 0,5 %, 1 %, 2 % and 5 % of the initial height of the sample and notification of the corresponding temperatures: T0,5, T1, T2 and T5. In this paper, only T0,5 values are presented, as usually in the refractory material data sheet.

The estimation of the main effect on signal values of each parameter (in reality twice the amount) was realised by subtracting the mean of run values at level −1 from the mean of run values at level +1. For example, for the effect of parameter X1 in the study of dense materials – "extraction" with level −1/level +1 = "corner/middle" – the results of runs 1, 4, 6 and 7 were averaged, from which the averaged results of runs 2, 3, 5 and 8 were subtracted. The following graphs show the estimated values of main effects on signal values of parameters investigated for the refractory materials tested. For better convenience, the results presented here are restricted to only T0,5 measurement, which is moreover the most current value on material data sheets.

On the basis of all the RuL temperature obtained, the Tab. 6 presents the most significant parameters for HA75. The criterion used to judge the significance degree of the factors is based on the estimation of the statistical significance level or "p-value". Indeed, factor with p-value less than 5 % will be considered as significant.

After analysis of these results, one can do the following observations:

- Loading is particularly significant for dense materials, excepting for pre-fired MCC75;
- The factor height has real impact whatever the material tested;
- Shaping direction only affects shaped materials;
- The position of thermocouple seems to largely influence pre-fired MCC75;

Tab. 4 T0,5 RuL values for HA75 (l.) and LWI35 (r.)*

HA75	
T_0,5 Values [°C]	
Reference	1504,0
1	1506,5
2	1478,0
3	1486,5
4	1396,0
5	1411,5
6	1468,0
7	1496,0
8	1480,5

LWI35	
T_0,5 Values [°C]	
Reference	1209,5
1	1203,5
2	1219,5
3	1112,0
4	1082,5
5	1158,0
6	1130,5
7	1181,0
8	1134,5

- To a lesser extent, the diameter only affects unshaped materials;
- To a lesser extent, the factor grinding has a quite global effect.

Tab. 5 T0,5 RuL values for unfired (l.) and fired MCC75 (r.)*

MCC75 Unfired	
T_0,5 Values [°C]	
Reference	1415,5
1	1415,0
2	1412,0
3	1411,5
4	1422,0
5	1416,0
6	1418,5
7	1421,0
8	1421,0
9	1421,5
10	1418,0
11	1418,0
12	1425,5

MCC75 Pre-Fired	
T_0,5 Values [°C]	
Reference	1414,0
1	1418,0
2	1411,0
3	1409,5
4	1416,5
5	1419,0
6	1416,5
7	1416,0
8	1424,0
9	1418,0
10	1415,0
11	1416,0
12	1134,5

*Reference tests follow the specifications stated in the standard

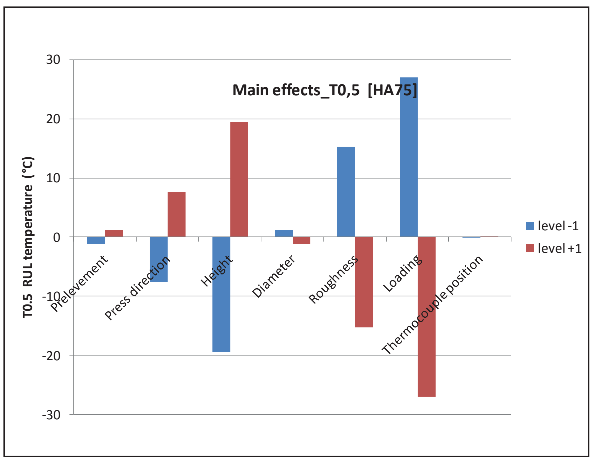

Fig. 1 T0,5 RuL values for HA75

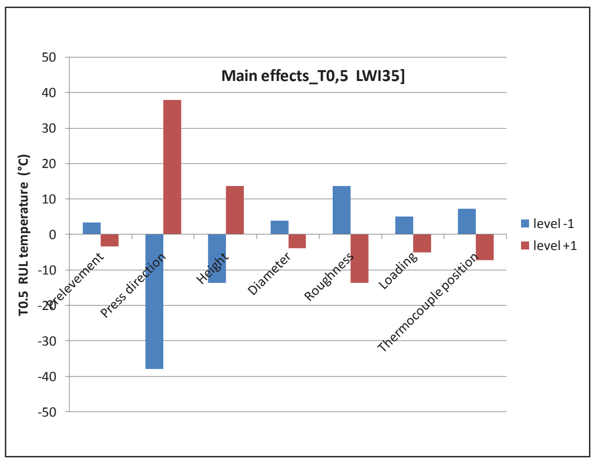

Fig. 2 T0,5 RuL values for LWI35

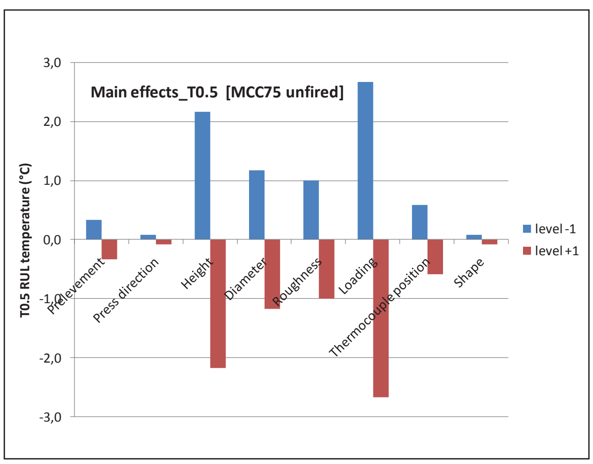

Fig. 3 T0,5 RuL values for unfired MCC75

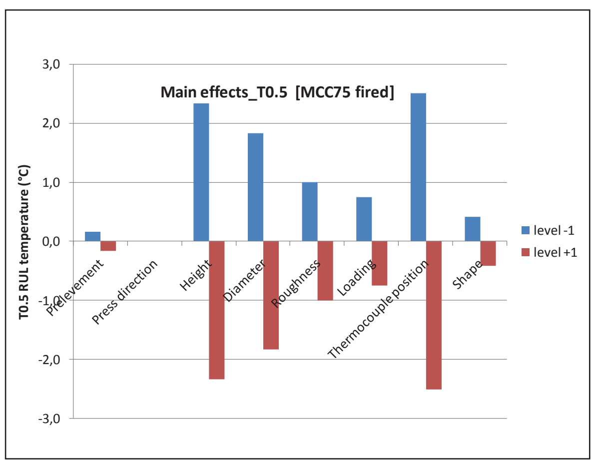

Fig. 4 T0,5 RuL values for pre-fired MCC75

Tab. 6 Most significant parameters on signal values

		Most Significant Parameters			
		LWI35	HA75	Unfired MCC75	Pre-Fired MCC75
Intensity of effect	+++	Shaping direction	Loading	Loading	Position thermocouple
	++	Height	Shaping direction	Height	Height
	+	Loading	Height	Grinding	Diameter
		Grinding		Diameter	Grinding

4 Appraisal of the influencing factors

Phase 1 has resulted in determining the potential factors that can influence data of the testing standard. Phase 2 is now aiming at quantifying the impact of the parameters selected from Phase 1. By the mean of fractional design involving several RTD performers, the aim is two-fold:

- Evaluate precisely the effects and possible interactions of the influencing factors;
- Find/check the factor values which are the most favourable for the practice and/or conduct to the best repeatability and reproducibility.

The same materials as in phase 1 have been investigated, but here within a limited Round Robin Test:

- A dense shaped refractory material (HA75);
- An insulating shaped refractory material (LWI35);
- A dense unshaped refractory material, pre-fired at 800 °C (MCC75).

4.1 Choice of the design plans

For each type of material, a fractional design plan of 8 runs is realized in order to evaluate four 2-level-factors, which will be presented next paragraph. For every combination of factors, and for all types of materials, a repeatability test will be realized by the mean of 3 test-pieces per experiment. In fact, for every experiment, three test-pieces will be extracted from the same initial brick.

The following testing parameters (factors) have been investigated:

Loading: Effect of the loading stress applied on the test-piece during the process.

The objective is to investigate the impact of a differential in loading, potentially due to the mistaken omission/addition of a mass in the device of mass balancing. This also can be seen as a research of more flexibility in the step of mass balancing. The standardized stress to apply is 0,2 MPa ± 2 %. *(Here, the associated levels are "0,2 MPa – 5 %" (level –1) & "0,2 MPa + 5%" (level +1)).*

Grinding: Effect of the roughness of upper and lower faces of the test-piece.

This parameter aims at the evaluation the necessity of grinding/refining operation of the test-piece. The possible suppression of this step could offer a considerable gain in time for the realization of the RuL test. According to the standard, measurements of the height at any two points shall not differ by more than 0,2 mm.

(Here, the associated levels are "grinding" (level –1) and "no grinding" (level +1).

Note: for the level "no grinding", it could happen that the faces after sawing are still conform to standard specifications, so an additional operation is carried out to artificially move away from standards.)

Position of thermocouple: Effect of the position of the central thermocouple.

It is required to measure the temperature "inside" the test-piece in order to identify the different RuL temperatures. The goal is to quantify the impact of a deviation in position of its junction from the mid-point of the test-piece. The normalized position is the geometric centre of the test-piece.

(Here, the associated levels are "centre" (level –1) and "centre –10 mm" (level +1)).

Platinum: Effect of the platinum sheet between test-piece and alumina discs.

This parameter permits to evaluate the effect of the presence of the platinum sheets between the test-piece and up and down alumina discs.

The standard specifies that such a sheet (in standard platinum or 10 % rhodium/platinum) is required in the event of a potential chemical reaction between test-piece and alumina discs.

(Here, the associated levels are "sheet" (level –1) and "no sheet" (level +1).

Note: this parameter has not been evaluated during phase 1, due to some problems of delivery capability.)

Height: Effect of the height of the sample.

The question of the height of the test-piece has to be considered insofar as the lower the height the less matter you use. It precisely goes in the sense of sustainable development. The normalized height is 50 ± 0,5 mm.

(Here, the associated levels are "50 mm" (level –1) and "30 mm" (level +1)).

Shaping direction: Effect of the casting/pressing direction in the test-piece.

The solicitation, which affects a cast lining may have different impact, depending on the orientation of the casting towards the direction of the solicitation. Indeed, the position of the axis of the cylindrical test-piece required for the test has to be considered towards the casting direction.

(Here, the associated levels are "axis parallel to casting direction" (level –1) and "axis perpendicular to casting direction" (level +1)).

Note: for shaped materials, the parameter "Shaping direction" will not be further investigated because of the solicitation of the bricks in majority along their pressing direction).

The parameter "diameter" has been deleted because of the required investment of specific drills, reminding that the first goal of the project is to simplify the using of the existing standard, and not engendering supplementary requirements.

"Height" and "grinding" are 2 of the 3 more significant parameters which are common between pre-fired and unfired unshaped materials. In fact, it could be pertinent to investigate, for unshaped materials, these 2 common factors, in complement of "loading" (unfired castable), "position thermocouple" (fired castable) and the parameter of "platinum foil" which could have not been investigated in the previous step of the study (phase 1). So, 5 parameters would have to be included in the next design plan dedicated to unshaped stabilized materials. As it has been judiciously decided to limit to 4 the number of parameters for design plan. A part of the parameters (all except the "platinum foil") will be displaced to the design plan dedicated to dense shaped materials and vice versa. The reason is that dense shaped material is reasonably considered as behaviouraly similar to dense stabilized unshaped materials. The following table summarizes the retained factors for each material studied.

Tab. 7 Parameters to investigate

Parameters to Investigate		
LWI35	**HA75**	**Unfired MCC75**
Platinum sheet	Loading	Shaping direction
Height	Position thermocouple	Height
Loading	Grinding	Grinding
Grinding	Platinum sheet	Platinum sheet

Tab. 8 Levels associated with HA75 parameters

N°	Factor	Level –1	Level +1
X1	Loading	Nominal –10 %	Nominal +10 %
X2	TC	Center	Center –10 mm
X3	Grinding	Yes	No
X4	PLAT	Yes	No

Platinum sheet: PLAT
Thermocouple position: TC

Tab. 9 Most significant parameters for each lab

Laboratory	To	T0,5	T1	T2	T5
	Signal Effect				
LAB 1	TC* Load	Roughness Load	Roughness Load		Roughness Load TC
LAB 2		PLAT	PLAT	out of range of testing device	
LAB 3	PLAT PLAT* TC			Load	Load
LAB 4	Load	Load Roughness	Load Roughness	Load	Load

Platinum sheet: PLAT
Thermocouple position: TC

Tab. 10 Most significant parameters for all labs together

Laboratory	To	T05	T1	T2	T5
	Signal Effect				
LAB 3 Lab 4	PLAT Grinding Load	Load Grinding	Load	Load	Load
LAB 1 LAB 3 LAB 4	PLAT Grinding	Load Grinding	Load Grinding	Load	Load Grinding
LAB 1 LAB 2 LAB 3 LAB 4	TC TC* Load	PLAT Load	PLAT Load	Load	Load Grinding

Platinum sheet: PLAT
Thermocouple position: TC

4.2 Results

The first results presented – which come from 4 different testing laboratories – refer to analysis of HA75. An estimation of the main effect on signal of each parameter has been realised, as in preceding step, but also an estimation of the dispersion values related to each combination of factors tested. Tab. 8 details the levels associated with each parameter investigated within the study of HA75.

Tab. 9 presents the signal influence of the investigated factors in taking into account each laboratory separately.

One can observe that load factor seems to be significant and that, in a lesser extent, grinding factor may be considered up to T1. One also can note that the results are not so homogeneous and clear than expected, in the sense that some parameters as platinum sheet for example have not the same degree of influence for all partners. Concerning the factor of roughness, the heterogeneity of the results can be partially explained by the fact that the level +1 (no grinding) has not been quantified, i.e. the degree of deviation of surface quality from the referenced standards is not necessarily the same for all partners.

Tab. 10 presents the signal influence of the investigated factors in considering all laboratories as a single entity, i.e. the origin of the results is implemented within the statistical analysis via a new artificial parameter called "laboratory". The 1st line concerns only 2 labs, while each succeeding line concerns 1 lab more, the 4th and last line concerning the results from all the 4 labs. The interpretation of data from Tab. 10 shows that load factor seems to be significant for all the RuL temperatures, which has already been observed within the lab by lab previous analysis. Furthermore, the first 2 lines of the table underline an important influence of the grinding parameter, but which disappears as soon as the results of the 4th and last lab are integrated with existing first data.

If one focuses on results for only T0,5, as well as loading, the factor of platinum sheet is significant on signal values. It means that, a particular attention has to be paid during the application of the load on the testing device. But also, the use of a platinum sheet (recommended by the standard if a chemical reaction between the test-piece and the alumina discs is assumed) can exert much influence on RuL temperatures, especially T0,5 and even T1.

After having evaluated precisely the effects of the influencing factors for HA75, finding/checking the factor values which conduct to

Tab. 11 Dispersion* values for HA75 T0,5 results

	Run 7	Run 1	Run 3	Run 8	Run 2	Run 4	Run 6	Run 5
Mean	1415,8	1439,2	1415,8	1457,0	1473,1	1462,5	1468,0	1440,9
Reproducibility	37,6	33,8	53,3	34,1	53,9	29,9	42,8	49,1
Reproducibility [%]	2,66	2,28	3,77	2,34	3,66	2,04	2,91	3,41
Repeatability	36,7	26,0	43,3	19,0	36,1	13,5	29,1	42,0
Repeatability [%)	2,60	1,81	3,06	1,30	2,45	0,93	1,98	2,91

RUN N°	Load	Platinum Sheet	Roughness	Thermocouple Position
7	Nominal +5 %	Yes	No grinding	Center –10 mm
1	Nominal +5 %	Yes	Grinding	Center
3	Nominal +5 %	No	No grinding	Center
8	Nominal +5 %	No	Grinding	Center –10 mm
2	Nominal –5 %	No	Grinding	Center
4	Nominal –5 %	No	No grinding	Center –10 mm
6	Nominal –5 %	Yes	Grinding	Center –10 mm
5	Nominal –5 %	Yes	No grinding	Center

Best repeatability/reproducibility
Worst repeatability/reproducibility

* Repeatability/reproducibility standard deviation (S_r/S_R) or repeatability/reproducibility interval (r/R with r = 2,8 S_r)

Tab. 12 Noise effects concerning HA75

Level +1	+10 %	NO	NO	–10 mm
Level –1	–10 %	YES	YES	0 mm
	Load	**PLAT**	**Roughness**	**TC**
r-average at level +1	31,25	27,96	33,89	24,58
r-average at level –1	30,16	33,44	27,52	36,83
Effect on r	0,55	–2,74	3,18	–6,13
R-average at level +1	39,71	42,81	42,48	36,10
R-average at level –1	43,91	40,82	41,15	47,53
Effect on R	–2,10	1,00	0,67	–5,72

Platinum sheet: PLAT
Thermocouple position: TC

the best repeatability/reproducibility (and are the most favourable for the practice) remains to be defined. For information, the Tab. 11 shows the dispersion values for T0,5 data.

As regards to statistical analysis of T0,5 measurement values, it is observable that combination N° 3 and combination N° 4 correspond to respectively the worst and the best repeatability/reproducibility values. A detailed examination of the factor levels involved in the 2 combinations would indicate that the position of thermocouple conditions the degree of dispersion of results (if it is considered that some or other levels of loading leads to equivalent dispersion values). Such a result seems to be surprising (almost non-relevant) regarding the fact that a deviation of the thermocouple position ensures the best dispersion, while a central position – that would have seemed to be the best suited – gives the worst one! The treatment of all RuL temperatures confirms the results extracted from T0,5 analysis.

(One has to keep in mind that all the results obtained come from a fractional design plan, so it means that the analysis could be slightly distorted due to the voluntarily use of only a fraction of all the possible combinations of factors. The runs here selected (N° 3 and N° 4) are not necessarily the optimum of the corresponding complete factorial design plan).

Furthermore, the 4 highest values of dispersion are very largely observed in the case of test-pieces with no surface preparation (no grinding process: 3 runs versus 1) and for a standardized configuration of the thermocouple (at the centre of test-piece: 3 runs versus 1). It has to be noted that "grinding levels" are not quantified, as it is the case for the levels of each other testing parameter.

The analysis of the noise effect of each factor, i.e. the influence of each parameter on the dispersion of the results is also performed. The process is exactly the same used for the analysis of the main effect on signal, with the difference that the software requires dispersion values (standard deviation) instead of signal values (RuL temperature). The Tab. 12 compiles the data obtained after statistical analysis. This computational treatment is useful to determine the factors, which have an influence on dispersion of data, especially the levels which ensure the smallest dispersion of results.

On the basis of the results presented on Tab. 12, one can note a significant noise effect for the position of thermocouple: it appears that level (+1) i.e. a deviation in position engenders a better dispersion of data, whether in terms of repeatability or in terms of reproducibility. In a lesser extent, roughness and use of platinum sheet affect the dispersion values. Indeed, the step of grinding as well as the non-using of platinum sheet allow an improvement of repeatability standard deviation.

5 Conclusion

The presented study has for goal to investigate the existing RuL testing methods, in order to suggest upgrading of related EN standards in focusing on quality improvement, cost reduction, convenience in use and time saving. The focus has been on the quantification of the effect of the more relevant parameters implied in RuL testing, by means of fractional factorial design plan. The aim is to improve repeatability and reproducibility of the data, and to assess the relevance of the specified tolerances of the standards.

The last results confirm the great influence of loading and platinum sheet (up to T1) on signal values. The high range of the investigated loading values (±10 %) compared to the standardized range (±2 %) allow us to maintain the prescription of the standard. In the event of use of platinum sheet, one has to keep in mind that the results may be different from those without sheet configuration.

In terms of noise effect, it appears that position of thermocouple considerably affects both repeatability and reproducibility of the data.

As surprising as it may sound, it is the non-centered configuration, which reduces dispersion of data. In a lesser extent, it seems that grinding process as well as non-use of platinum sheet engenders an improvement of dispersion.

In the current state of this research, one can suggest that the current specification of the standards do not require any modification for the usual testing of dense shaped materials. However, an additional note may underline the non-negligible effect of use of platinum sheet, in terms of both signal deviation and dispersion downgrading.

Acknowledgments

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