6.780 term project

Improvement in Mechanoluminescence Intensity of $Ca_2Al_2SiO_7$: Ce by the Statistical Approach

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Introduction

What is Mechanoluminescence?

Mechano = apply force/stress

Luminescence = light emission

Mechanoluminescence refers to the light emission from a material induced by stress that caused deformation or fracture of solids.

Background

Mechanoluminescence phenomena exist in silica and quartz, sugar, rocks, alkali halide, II-VI compounds and polymer crystals. However, the mechanoluminescence intensities of these existing materials are not strong enough for practical usage.

Recently it was found that rare-earth-doped oxides emit comparatively intensive visible light when stress is applied to these oxides, e.g. Ce^{2+} (blue), Eu^{2+} (green), etc

Background

Applications of mechanoluminescence materials

Stress sensor to visualize stress distribution in macro and micro-scale

- New ML type stress sensors
- Ultrafine spherical particles
- Plasma displays
- Novel mechano-display
- Poison-free lighting

Background

- Complex of mechanoluminescence design and optimization. Multiple factors and their interactions will lead to numerous experiment runs and tremendous costs.
- Statistical approach will dramatically reduce the size of experiment runs.
- The price to pay is a decrease in the recognition of the significance of the interactions between main effects.
- High level of the process knowledge is essential for the judgement

Experimental procedures

Design of experiment – mixed $2^1 3^7$ factorial design

One factor at two levels, seven factors at three levels.

Factors		Levels		
A	Atmosphere	Ar	Ar+H_2(5%)	--
B	Temperature(°C)	1200	1300	1400
C	Sintering time (h)	1	3	5
D	CeO_2 (mol)	0.00001	0.00005	0.00010
E	Al_2O_3 (mol)	0.009	0.010	0.011
F	SiO_2 (mol)	0.009	0.010	0.011
G	$CaCO_3$ (mol)	0.018	0.020	0.022
H	H_3BO_3 (mol)	0.001	0.002	0.003

Experimental procedures

L_{18} orthogonal array design and test result

#	A	B	C	D	E	F	G	H	Cps
1	0	0	0	0	0	0	0	0	44
2	0	0	1	1	1	1	1	1	179
3	0	0	2	2	2	2	2	2	1403
4	0	1	0	0	1	1	2	2	273
5	0	1	1	1	2	2	0	0	8379
6	0	1	2	2	0	0	1	1	293
7	0	2	0	1	0	2	1	2	2257
8	0	2	1	2	1	0	2	0	154
9	0	2	2	0	2	1	0	1	7343
10	1	0	0	2	2	1	1	0	192
11	1	0	1	0	0	2	2	1	123
12	1	0	2	1	1	0	0	2	4224
13	1	1	0	1	2	0	2	1	270
14	1	1	1	2	0	1	0	2	7782
15	1	1	2	0	1	2	1	0	1740
16	1	2	0	2	1	2	0	1	13324
17	1	2	1	0	2	0	1	2	792
18	1	2	2	1	0	1	2	0	288

Adapted from D. C. Montgomery, *Design and Analysis of Experiments*, 4th ed., John Wiley Sons, New York 1997, p. 625-641

Experimental procedures

ANOVA table of mechanoluminescence intensity

The amount of Ca^{2+} and Si^{4+} cations are most important factors !

ANOVA table

Source	S.S.	d.f	M.S.	F	P(F)
B. temp	283.8	2	141.9	12.9	0.01
D. CeO_2	138.8	2	69.4	6.3	0.04
E. Al_2O_3	79.4	2	39.71	3.6	0.1
F. SiO_2	383.6	2	191.8	17.4	0.006
G. $CaCO_3$	1537	2	768.75	69.7	0
H. H_3BO_3	96.28	2	48.14	4.4	0.08
Error	55.17	2	11.03		
Total	2576	17			
R^2	98%				

Crystal structure of $Ca_2Al_2SiO_7$

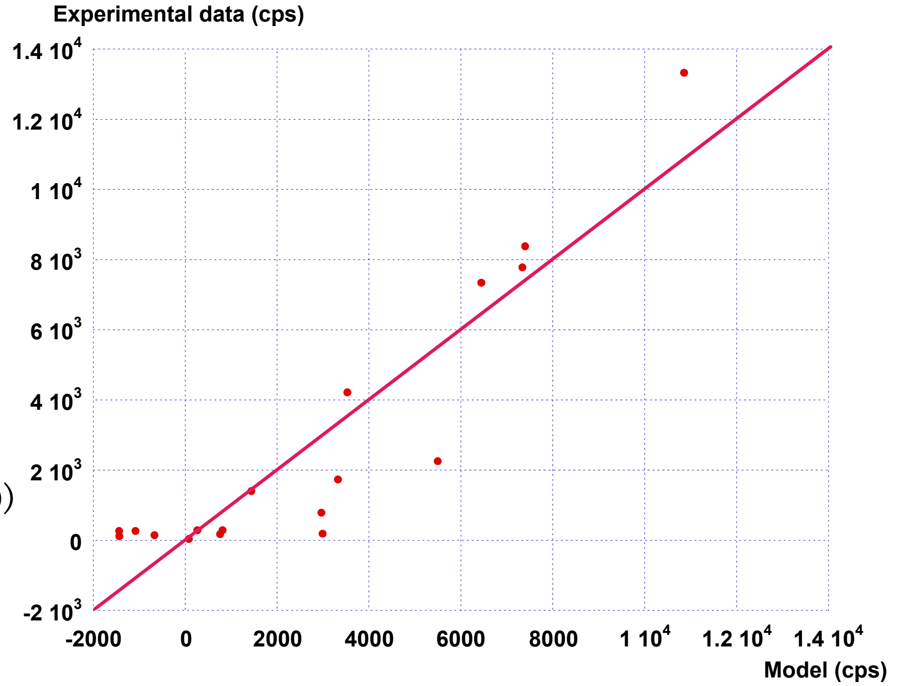
S. I. Kubota, et al, J. Alloys Compd. 283, 95 (1999)

Experimental procedures

Individual effect of each factor

Effect	Level 0	Level 1	Level 2
A	-467.6	467.4	--
B	-1696.8	396.8	1300
C	1.66	175.5	177.5
D	-1006.8	-126.5	1133
E	-928.2	589.7	338.2
F	-1763.2	-48.8	1811.7
G	4123.3	-1816.2	-2307.5
H	-925.5	862.7	62.5

$$cps = 1000 \times (0.0799 + A \times 0.9351 + B \times 1.4989 - C \times 0.0896 + D \times 1.0699 + E \times 0.6332 + F \times 1.7874 - G \times 3.2154 + H \times 0.4940)$$



First order linear model

Conclusion

- Orders of magnitude increase in the mechanoluminescence intensity
- The mechanoluminescence intensity is sensitive to the crystal structure, which is highly influenced by the amount of Ca & Si cations in the crystal
- First order linear model fits the experimental data well