Analysis of Particle Size and XRD in ZnO-based Varistor

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ZnO varistors doped with Bi_2O_3, Sb_2O_3, CoO, MnO_2 and Cr_2O_3 were prepared separately by two mixing processes: conventional and masterbatching mixed-oxide process method. After the mixing process, the ZnO-based varistor powders were milled using planetary ball mill at 200, 300, 400 and 500 rpm. Particle size analysis and XRD analysis were used to study the differences after milling process in ZnO-based varistor powder. The particle sizes of varistor powder were reduced and new compound encountered after milling process.

MATERIALS AND METHOD

The detail of the varistor powder compositions is given in table I.

Table 1 The composition of the varistor

	ZnO	Sb_2O_3	Bi_2O_3	CoO	MnO_2	Cr_2O_3
mol %	97	1	0.5	0.5	0.5	0.5

For conventional route, the powder was weighting according to the mol% and mixed together. In the masterbatching mixing route, the same compositions of varistor powder prepared in two levels to get the final formula $A_{0.03}B_{0.97}$ where A is trace element and B is ZnO. At the first level, the powders contained 70 mol% of ZnO and 30 mol% of trace element. In order to get the final formula, 10 mol% from level 1 is added to 0.90 mol% ZnO at the second level.

The composition was prepared by milling a mixture in varies speed; 200, 300, 400 and 500 rpm. Particle analysis was done using particle analyzer (Malvern Mastersizer 2000)The mixed powders were calcined at 750°C for 2 hours. Then the powder was pelleted before sintered at 1200°C for 2 hour. The crystalline phases of the samples were identified by Lab X XRD-600 Shimadzu.

RESULTS AND DISCUSSIONS

Figure 1 represents the mean d (0.5) for each powder. The mean size for pure ZnO is 1.843 µm. After the pure ZnO weighting and mixed according to the mol% showed in table 1 the mean size was a little bit change which is d (0.5) = 2.556 µm. For both mixing route, the mean size increase with the increment of the milling speed. Seemingly, the additive and ZnO react therefore the agglomerate formed even thought the powder was diluted in ethanol before characterized. The agglomerate increases the mean size of varistor powder.

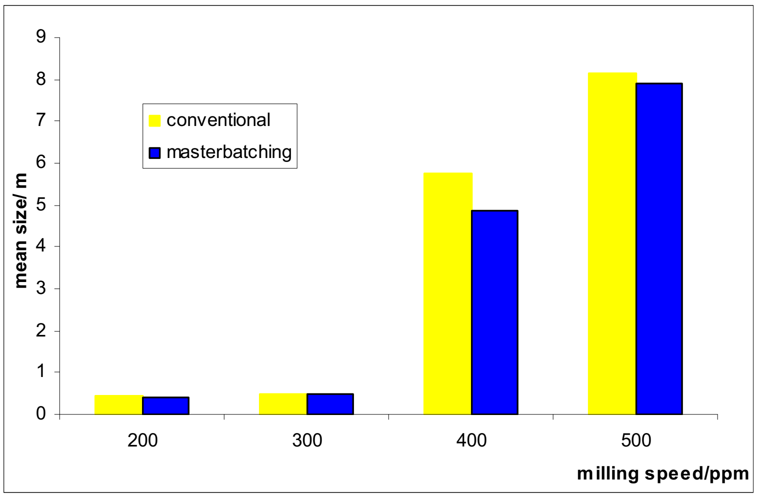

Figure 1: Result for particle analysis

Figure 2 shows the XRD pattern for varistor powders mixed using conventional mixing route. The main phase of the entire sample is confirmed by PCPDF database. The phases found are ZnO (No. 36-1451), Bi_2O_3 (No. 52-1007) CoCrO4 (No. 1-1082). It shows that, after sintering process new compound formed. The XRD analysis for both mixing routes, show the same pattern for each milling speed but the intensity change.

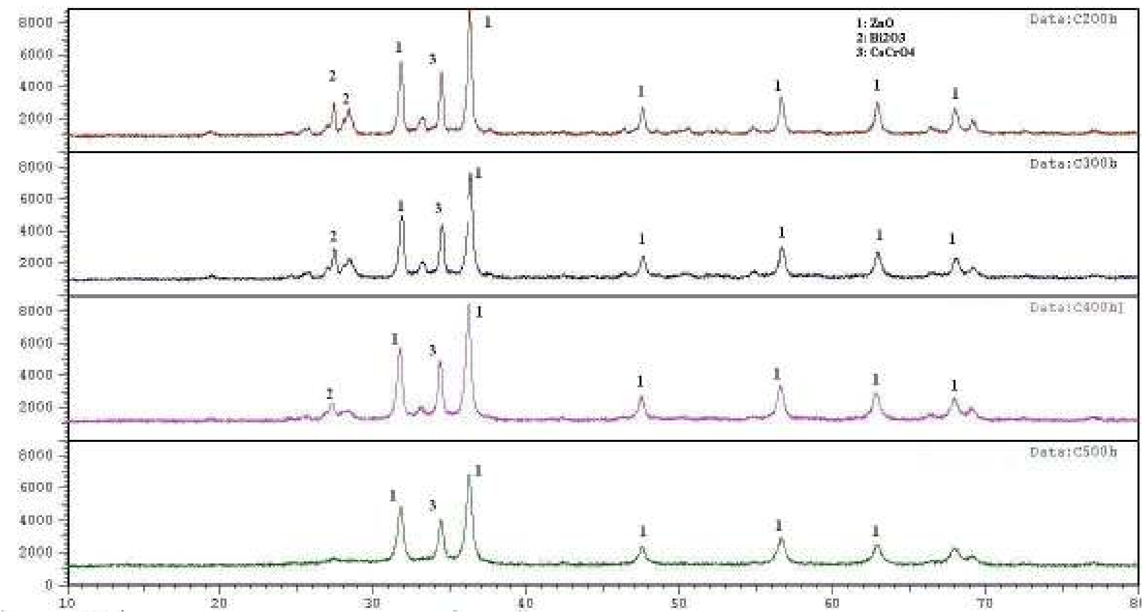

Figure 2: XRD pattern for sample mixing using conventional mixing technique

Figure 3 shows the XRD pattern for masterbatching mixing route varistor powders For masterbatching mixing route, the varistor powder showed the phase ZnO, CoCrO4 (No. 1-1082) and $CrMnO_4$. Those pattern belong to 2 space group which are P63mc (ZnO) and Fd-3m ($Co_{2.33}Sb_{0.67}O_4$ and $CrMn_{1.5}0_4$). This result explains that the sintered pellet had been reacted to form at least 2 phases that belong to the both of the space group. The Bi_2O_3 which is unable to trace by the database could be substitute in the same site of Sb in the structure because both of the elements are in the same group.

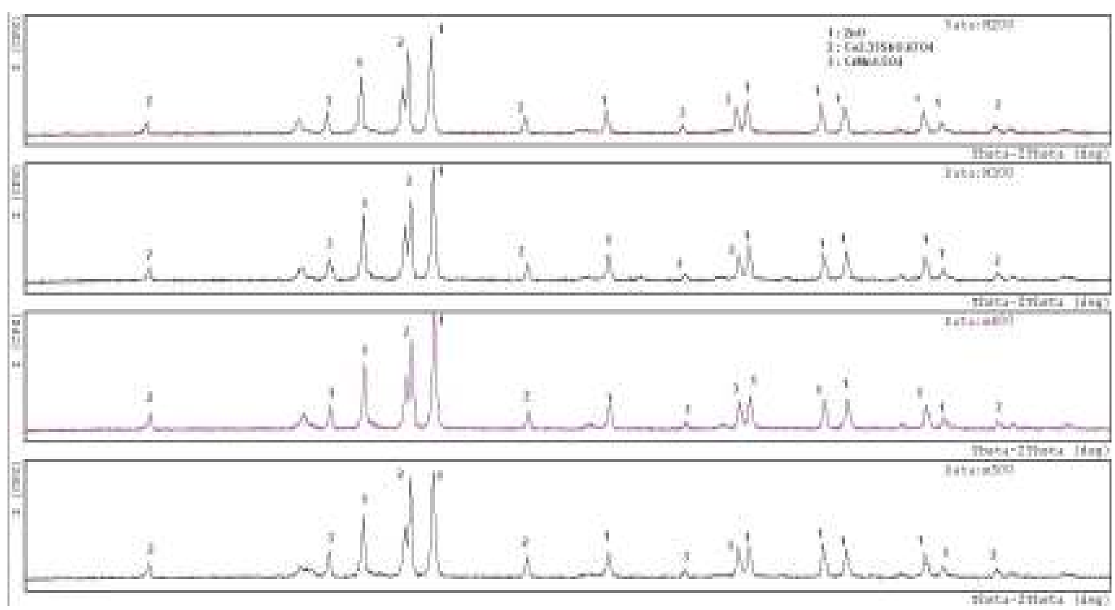
Figure 3: XRD pattern for sample mixing using masterbatching mixing technique

CONCLUSION

In order to obtain the fine-grained and uniform microstructure, the preparation of varistor powder needs to control. The ZnO - Bi_2O_3 - Sb_2O_3 - MnO_2 - Cr_2O_3 -CoO based varistor is produced by conventional and masterbatching mixing route. The mean size of varistor powder milled at 200 and 300 ppm where fine making them potentially attractive for further investigation and evaluation. Meanwhile, the XRD pattern showed the sintering process react to form a good varistor for each milling speed for both mixing route.

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