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IMPROVED DENSITY CONTROL
OF MOLDED DESICCANTS

EP 04119931, Topical Report

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**Kansas City
Division**

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IMPROVED DENSITY CONTROL OF MOLDED DESICCANTS

BDX-613-910 (Rev.), UNCLASSIFIED Topical Report, Published August 1973

Prepared by J. M. Conway, D/814 under EP04119931

A process was initiated which evaluated the effects of using flexible tooling in the cold pressing of molded desiccant billets. Significant improvements in billet uniformity resulted from the use of the flexible tooling regardless of billet thickness or mold loading techniques.

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SUMMARY

A molding process designed to obtain better density uniformity in parts compression molded from poorly flowing compounds has been achieved. The process makes use of a flexible rubber pad, inserted in a compression mold between the punch and charged material. The flexible pad deforms when a load is applied, thus ensuring that uniform pressure is applied across the irregular pad part interface. The molding compound used was a mix having flow characteristics comparable to damp sand and made from Davison 3A molecular sieves and an epoxy resin matrix.

Two formulations of a desiccant mix were evaluated: one consisted of 18.3 percent epoxy resin; the other had 30 percent resin. Of primary interest was how the density uniformity of these desiccant billets compared to the density uniformity of billets molded using the standard process. Billet thickness and mold charging technique were compared.

Density variations were determined using three measurement techniques: density times thickness (DXT), isodensitracer (IDT), and microdensitometer (MDT). DXT and MDT tests were used to scan density variations along a desired path, usually the surface of a billet. IDT charts were used when the density distribution in a cross-section of a billet was desired. Density testing was performed on the desiccant billets in directions parallel and perpendicular to the molding pressure. Three conclusions were derived from the test data. The use of flexible tooling in the compression pressing of molded support desiccant results in parts having a much more uniform density than those molded from standard tooling. The density uniformity achieved by the use of the rubber pad is not significantly affected by variations in billet thickness between 0.4 and 0.9 inch (10 and 23 mm); however, the density uniformity in billets molded by the standard process deteriorates as the part thickness decreases. The flexible tooling reduces the adverse affects of such processing problems as uneven charging of the mix in the mold cavity and agglomerates that sometimes occur in the mix.

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DISCUSSION

SCOPE AND PURPOSE

Currently, a Y10 part is machined from molded desiccant billets 4.3 inches (110 mm) in diameter approximately 0.7 inches (18 mm) thick. The desiccant billets are fabricated from a mix process according to MS9922025 and room temperature pressed on a compression press. The billets are then machined into the final configuration and the diameter and thickness are reduced to 4.0 and 0.3 inches (102 and 8 mm) respectively.

The objective of this study was to evaluate a molding process to determine if better density uniformity in molded support desiccant billets could be achieved. Such a process would result in stronger parts, more consistent and reliable test data, and substantial cost savings. By maintaining density uniformity in molding thinner billets during production, material and machining time could be conserved.

ACTIVITY

Preparation of the Desiccant Billets and Test Samples

Two batches of Davison 3A molecular sieves were mixed in a Hobart vertical cutter-mixer, as described in MS9922025 and SSP65750, using the following formulations.

X10 Formulation (SSP65750)

Davison 3A Molecular Sieves	1000 pbw
Epon 871 Epoxy Resin	300 pbw
Shell Curing Agent Z	37.2 pbw

Y10 Formulation (MS9922025)

Davison 3A Molecular Sieves	1000 pbw
Epon 871 Epoxy Resin	160 pbw
Epon 828 Epoxy Resin	40 pbw
Shell Curing Agent Z	24.8 pbw

After mixing, the two lots were pressed at room temperature for 3 minutes at pressures of 2500 psi (17.2 MPa) for the X10 material and 4000 psi (27.6 MPa) for the Y10 material. A total of 29 billets (Numbers 1294 through

1322), 5 inches (130 mm) in diameter and having thicknesses of about 0.5 and 1 inch (13 and 25 mm) were pressed. About half of the billets were pressed using only the standard compression mold tooling, while the remainder were pressed using a General Electric RTV 630 rubber insert in the same mold. Figures 1 and 2 illustrate the tooling used in the new molding process and the desiccant billets produced by each process.

The RTV insert was cast in the mold used to press the desiccant billet and cured for 16 hours at room temperature. This process yielded an inset approximately 5 inches (130 mm) in diameter and 1 inch thick (25 mm), having a Shore A durometer hardness of 15. Because the room temperature cure left the RTV insert slightly tacky, it was necessary to place a circular cut of paper between the insert and the desiccant mix. The tooling is illustrated in Figure 3.

Six of the 29 billets (1297, 1304, 1309, 1310, 1321, and 1322) were used to determine if the flexible tooling technique could negate the adverse effects of uneven charging for both billet formulations and thicknesses. Uneven charging occurs when the desiccant mix is poured into the mold cavity with no attempt made to level the surface. A mechanical leveling device is used to provide even charging.

Uneven charging can drastically affect density uniformity and thus affect the physical properties in poorly flowing materials such as the desiccant resin mix. A poorly flowing mix resists movement when pressure is applied; when such a mix is charged unevenly, the punch contacts only high spots in the mix and the load is propagated to only a fraction of the mix.

The remainder of the processing involved the curing, machining, and activation of the billets. Immediately after pressing, the billets were cured at 135°C for 48 hours. The surfaces of the billets were then machined to make test samples for MDT, DXT, and IDT testing. After machining, the test specimens were activated at 175°C for 24 hours at a reduced pressure. The activated samples were then immediately heat-sealed in vapor barrier bags to prevent moisture absorption. A flow chart outlining the general procedure is given in Figure 4.

Density Times Thickness (DXT) Testing

DXT tests were performed to determine the variations in billet density that occur when the billets are scanned in directions shown in Figure 5. It should be noted the direction of X-ray penetration was parallel to the molding pressure while the direction of traverse was perpendicular to the molding pressure. DXT testing requires that samples have a uniform thickness so it was necessary to machine the desiccant billets to thicknesses of 0.4 and 0.9 inches (10 and

Figure 1. Conventional Compression Molding Tooling with Flexible Insert

PRESSED USING FLEXIBLE INSERT

PRESSED USING STANDARD METHOD

Figure 2. Billets Fabricated by the Two Molding Techniques

23 mm). During testing the samples are placed between an X-ray source and a detector which records the amount of X-rays emitted through the sample. The amount of X-rays that penetrates is a function of sample density and thickness. The product of density and thickness is recorded on a strip chart. Calibration curves are predetermined using step blocks made from material identical to that used in making the test samples. The X-ray source is directed through an aperture 0.25 inches (6. mm) in diameter so that the densities recorded are an average of the various densities that occur in a line of this width across the billet. DXT testing was used to determine billet densities from point to point and does not show cross-sectional area density distribution.

Figure 3. Cross Section of Charged Molding Using Rubber Insert

Microdensitometer (MDT) Testing

MDT testing is essentially the same as DXT testing as far as output data is concerned, but the experimental setup is different. A light source is placed under an X-ray radiograph of the sample and a photodetector measures and records the amount of light that passes through the film. The sensitivity of the MDT tester is much higher than the DXT tester since the aperture diameter of the light source is only 0.0005 inches (0.013 mm). This enables the microdensitometer to distinguish density variations caused by microscopic particles of comparable diameter. All billets that had been tested using the DXT method were also tested on the MDT. The directions of the scans were

Figure 4. Flow Chart for Density Uniformity Study

identical to those used for DXT testing (Figure 5). The data in Tables 1 and 2 represent an average of the DXT and MDT readings. Each value represents a percentage density variation that is an average of the two 90-degree scans. The data in the tables is separated according to charging technique, formulation, and billet thickness. It can be seen that in all cases the flexible tooling improves density uniformity compared to the standard process. The data also illustrates that uneven loading affects the density uniformity of billets fabricated from the standard process much more than billets molded using the flexible pad. This was especially true for the X10 formulation. One surprising result was that for both molding techniques, a higher density uniformity was achieved in billets of greater thickness.

Figure 5. Direction of DXT Scan of Test Specimen

In Figure 6 two typical MDT graphs illustrate the density uniformity of billets molded by the two processes. Billet 1306 was molded by conventional compression molding tooling, while Billet 1307 was pressed using the same tooling with the flexible pad. In the graphs there are local peaks and depressions which indicate high and low density inclusions. (For this study, these variations are not important and only indicate that some impurities are present in the desiccant mix.) Of interest, however, is the general slope of the line in the graph. The overall deviation from the horizontal represents a variation in the quantity DXT. Since specimens machined to a uniform thickness are used, the variation in the quantity DXT is known to be caused by a density variation in the sample.

Table 1. Percent Density Variation in Molded Support
Desiccant Discs, 5 Inches (130 mm) in Diameter and
0.4 Inch (10 mm) Thick

Mix Loading Technique	X10 Formulation		Y10 Formulation	
	Standard Tooling	Flexible Tooling	Standard Tooling	Flexible Tooling
Even	7.17	1.25	3.30	1.43
	6.35	0.72	4.04	1.66
			4.07	2.01
			3.19	3.11
			2.45	2.82
Average	<u>6.76</u>	<u>0.98</u>	<u>3.41</u>	<u>2.20</u>
Uneven	20.03	2.90	4.89	2.19
	11.90	0.57	2.12	0.84
Average	<u>15.96</u>	<u>1.75</u>	<u>3.50</u>	<u>1.51</u>

Table 2. Percent Density Variation in Molded Support
Desiccant Discs, 5 Inches (130 mm) in Diameter and
0.9 (23 mm) Inch Thick

Mix Loading Technique	X10 Formulation		Y10 Formulation	
	Standard Tooling	Flexible Tooling	Standard Tooling	Flexible Tooling
Even	0.54	0.78	1.05	1.30
	0.83	0.58	1.57	0.50
	0.82	0.58		
Average	<u>0.73</u>	<u>0.64</u>	<u>1.31</u>	<u>0.90</u>
Uneven	4.15	2.40		
	1.75	0.82		
Average	<u>2.90</u>	<u>1.61</u>

Figure 6. Microdensitometer Graphs of Two Billets

Isodensitracer (IDT) Testing

IDT is a step-scanning connected to a microdensitometer. The step-scanning capability enables the system to record density distribution of cross-sectional areas. The output consists of multicolored regions in which areas having identical densities are represented by one color on the graph. The accuracy of an IDT is comparable to that of a microdensitometer. IDT scans were perpendicular to the direction of the molding pressure and were used to scan the cross-sectional areas of the billets in a manner analogous to the direction of scan in Figure 7.

The density distribution in cylindrical compacts has been the subject of research by Train and Lewis ("Agglomeration of Solids by Compaction," Institution of Chemical Engineers Transactions, Volume 40, Number 9, 1962, p. 237). Studies involving the compaction of granular particles reveal the presence of a dense axial core near the bottom of the compact when cross

sections of the compact are examined. Figure 7 gives the density distribution in a cylindrical compact of magnesium carbonate powder. (The numbers shown are not densities but are unitless numbers which reflect the relative densities of regions in a compact.)

Cross-sectional areas in desiccant billets were scanned by an IDT to determine the density distributions. Desiccant billets molded using standard tooling had high density areas midway down in the compact. These areas, however, shifted horizontally depending upon mold charging technique. When the flexible pad was used, billets were uniform in density throughout.

Figure 7. Density Distribution in a Cylindrical Compact of Magnesium Carbonate at an Applied Pressure of 29000 psi (200 MPa)

Two IDT graphs (Figures 8 and 9) reveal the cross-sectional density distributions for billets molded by the two processes. Figure 9 shows the density distribution in a billet molded using the flexible insert. Normally on IDT graphs different colors or different shades of one color indicate areas of different densities. The areas on the IDT graphs near the edges are not actually density variations but are the result of X-ray scatter and are called fringe effects. False readings thus appear near the edges on both graphs. The density of the cross-section of a billet molded using the flexible pad is uniform.

The density distribution for the billet molded from the standard tooling (Figure 8) varies from 1150 to 1230 kg/m³. The 1090 kg/m³ value in Figure 8 is probably the result of fringe effects. The variations in densities that occur for the billet molded using the standard process occur horizontally, suggesting that the top surface of the molding compound was not completely level when charged despite the use of a mechanical leveling device. The use of the new process when pressing the desiccant billets would improve the density uniformity despite such processing variations.

ACCOMPLISHMENTS

Significant improvements in the density uniformity of molded support desiccant billets were achieved using the new molding process. Superior density uniformity in desiccant billets is maintained regardless of billet thickness when the new process is used.

Figure 8. Isodensitracer Sketch Showing Density Variation in Molded Desiccant Using Standard Tooling

Figure 9. Isodensitracer Sketch Showing Density Variation in Molded Desiccant Using Flexible Tooling