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Failure Investigation & Analyses of AISI D3 Dies used in Tableting Machines for Pharmaceutical Application

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Abstract. AISI D3 material finds wide use as Dies in Tableting Machines for Pharmaceutical applications owing to its good strength, toughness and moderate corrosion resistance combined with cost effectiveness. However, 10-15% of AISI D3 Dies used in rotary Tableting Machines were found to fail within 4-6 months of service resulting in frequent disruption of regular production and loss in overall productivity besides having potential long-term risks such as metal contamination in the output product.

Visual examination of the failed Die showed 180° crack across the top Die face with rust marks, but no such crack was found on the bottom face. Also, the crack on the top Die face had a jagged morphology and those cracks on the Outer Diameter (OD) surface oriented in the axial direction were found to be straight and sharp. Macro-examination of Die sectioned diametrically and perpendicular to the crack on the Die face revealed jagged axial cracks on the Internal Diameter (ID) surface. Cluster pitting marks centered around mid-height of the Die were observed on the ID surface and the cut open sections of the Die along the crack revealed extensive corrosion. While microstructural examination of samples taken on the ID surface showed presence of banded carbides oriented axially in tempered martensite matrix, the same examination carried out on samples taken on Die face revealed fine needle like carbides in tempered martensite matrix. Fractography analysis conducted in the cluster pitting zone on the ID surface clearly showed presence of micro-cracks having multiple points of origin with clear indications of propagation into the cross section of the Die. Scanning Electron Microscopy (SEM) carried out in the aforementioned zone near ID surface also showed the presence of multiple micro-cracks. The SEM-EDX analysis in the referred zone at specific locations in the vicinity of microcracks confirmed inadequate Chromium oxide content leading to lack of corrosion resistance in the material.

Based on the investigation & analyses and the detailed study of the Tableting Process in rotary Tableting Machines that revealed the nature of applied load to be cyclic, a comprehensive failure mechanism was arrived at, and it was inferred that the predominant mode of AISI D3 Die failure was Corrosion Fatigue. Finally, recommendation has been proposed to prevent such failures during service.

Introduction

Tableting Machine – An overview. The Tableting Machine also known as tablet compressing machine is used to compress pharmaceutical powder formulations into uniform shaped & sized tablets. There are two broad types of tablet compression machines, as follows:

- Single Punch Presses
- Rotary Presses

Single Punch tablet press is the simplest equipment, which uses a single set of tooling consisting of a Die and a pair of upper & lower punches, for manufacture of tablets. The compaction force on the fill material is executed only by the upper punch while the lower punch is stationery. The single punch tableting press generally produces 60-80 tablets / minute.

Rotary tablet press is a mechanical device, which has several tooling stations that rotate to compress the powder mixture into tablets of uniform size, shape, and weight. The compaction force on the fill material is executed both by the upper and lower punches resulting in the powder granules getting compressed in the middle. The rotary tablet press was primarily developed to increase the output of the Tableting Machine. The capacity of the equipment is determined by the rotation speed (RPM) and the number of stations on the press. Rotary Tableting Machines typically give an output between 10,000 and 200,000 tablets per minute.

Tableting Process. The basic principle involved in the Tableting Process is compression of powder / granules between upper & lower punches in a Die hole through application of uniform hydraulic pressure. Following are the main stages involved in the Tableting Process:

- Filling
- Metering
- Compression
- Ejection

The filling stage involves transfer of powder / granules to the machine Die cavity and the granulation is overfilled to ensure complete filling of the punch-Die cavity. The metering stage involves removal of excess granulation or powder from the Tableting machine. The compression stage, which involves bringing together upper & lower punches under pressure within the Die hole, forms the tablet. Finally, the ejection stage involves removal of formed tablet from the lower punch station. The scraper then pushes the formed tablets away from the Tableting Machine into the collection bin through discharge chute. The schematic of the Tableting Process is shown in Fig.1.

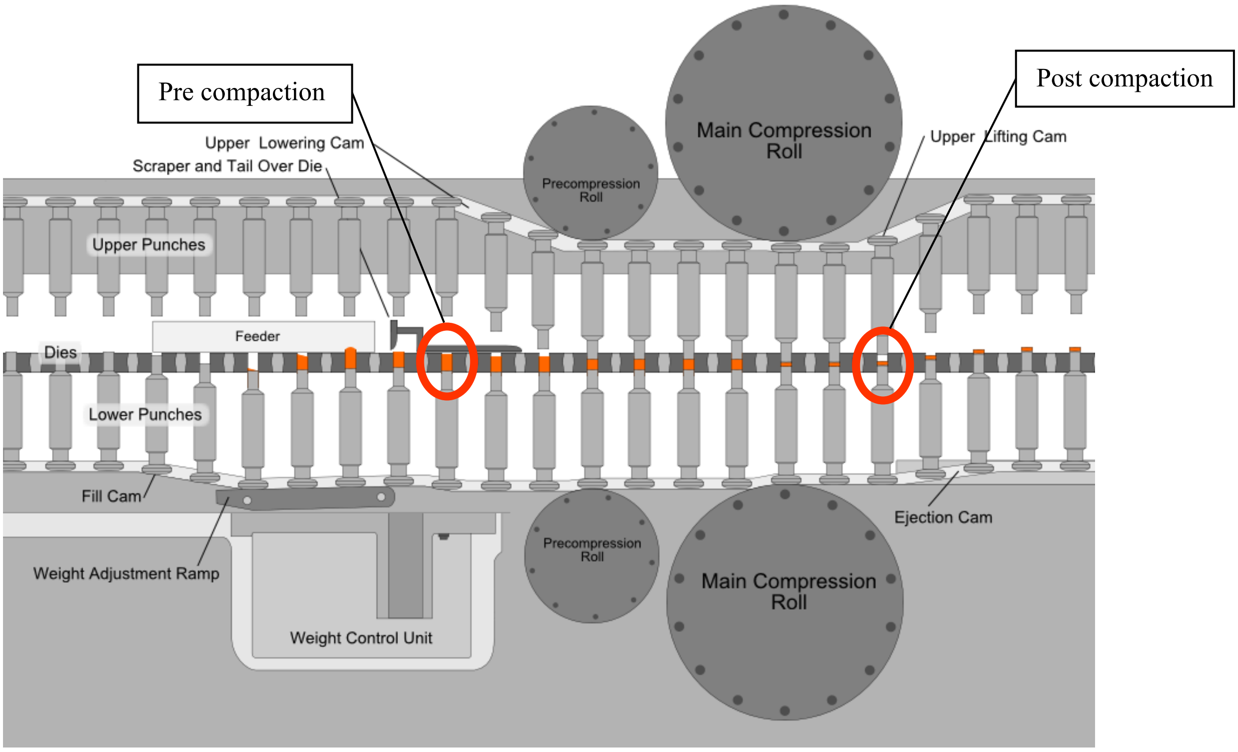

Fig. 1. Schematic of Tableting Process

Punch & Die System. In order to produce tablets, the punches move within the Die cavity thereby compressing the powder into tablets of desired shape, size and weight. The upper punch system, which is located on the upper section of the rotary system, moves vertically in and out of the Die bore. The lower punches, which are located on the lower section of the rotary system, move vertically but remain within the Die bore. Also, once compacted, the lower punch system enables ejection of the tablet from the Die hole. The sectional view of the elliptical Die are shown in Fig.2 below.

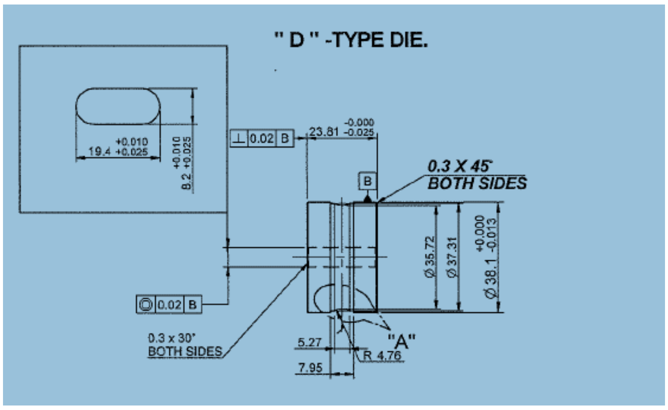

Fig.2. Sectional view of elliptical Die

The manufacturing sequence of the Dies involves cutting off the needed length from annealed rolled raw material stock of required diameter and rough machining the pieces to required dimensions followed by heat-treatment, which is generally a hardening & double tempering operation, to achieve a hardness of 60-64 HRC. Subsequently, the heat-treated pieces are subjected to grinding operation, Wire Electrical Discharge machining to form the central elleiptical or round hole and fine polishing before using them in rotary Tableting Machines. The details of the required chemistry and hardness are given below in Table 1 and that of inclusion rating of the steel is provided in Table. 2.

Table 1. Chemical composition and hardness of AISI D3 Dies

Composition		C	Mn	Si	S	P	Cr	Mo	Ni	V	W	Hardness, HRC
Specification AISI D3 (%)	Min	2.00	0.10	0.10			11.0					60
	Max	2.35	0.60	0.60	0.03	0.03	13.5		0.03	1.00	1.00	64
Round Die	Actual	2.26	0.35	0.37	0.01	0.012	11.83	0.13	0.11	0.09	0.12	62
Elliptical Die	Actual	2.10	0.35	0.38	0.01	0.014	11.81	0.12	0.11	0.09	0.11	62

Table 2. Inclusion Rating

Type	Sulphide		Alumina		Silicate		Oxides	
Avg Size	Thin	Thick	Thin	Thick	Thin	Thick	Thin	Thick
	0	0	0	0	0	0	1	0.5

Site Information

Service History. Cracking & breakage of Dies in Tableting Machines during regular operation after a service period of 4-6 months was reported in both round and elliptical Dies. About 10-15% of the Dies crack on a regular basis. The data collected was for the period October to December 2019.

Operating Parameters.
(a) Material being processed : Pharmaceutical formulation
(b) Applied Load : 75 – 80 KN
(c) Speed of the rotary Tableting Machine : 50 Revolutions per minute
(d) Hours of operation : 2 shifts (~ 16 hours / day)

Failure Signs. There are generally no prior indications or forewarnings before sudden cracking of the Dies resulting in abrupt stoppage of the rotary Tableting Machine.

Experimental

Two AISI D3 Dies, one each round and elliptical, were received for analysis. Both were visually examined followed by Macroscopic examination with Celestron Digital Microscope Model No. 44308 and digital photographs were recorded. The Dies were then cut sectioned perpendicular to the crack observed on the Die face using Wire Electrical Discharge Machining (WEDM) method. Macro-examination was conducted on the cut-sections besides Liquid Penetrant testing, which was performed in accordance with ASTM E-165 standard. Subsequently, the cut sections were subjected to minor load using a vise to enable breakage along the crack to examine the fractured Die surfaces.

Samples were taken for chemical analysis, determination of inclusion rating, microstructural examination, and SEM Analysis. Chemical Analysis of the Dies was conducted using Spectromaxx Model No. 1433941 and inclusion contents were determined in accordance with ASTM E-45 standard. For microstructural analysis, the specimens taken from both the Die faces and along the Inner Diameter of cut sectioned pieces were mounted in cold setting mold with plastic resin and metallographically polished. The polished surfaces were then etched with Marbles reagent and the etched surfaces were viewed under Meiji Inverted Optical Microscope Model No. IM 7200.

The fractured surfaces of both the Dies were subjected to macroscopic examination using Celestron Digital Microscope Model No. 44308 and microstructural examination at high magnification using Carl Zeiss Sigma Scanning Electron Microscope (SEM). Also, Energy dispersive X-ray Analysis (EDX) was carried out to determine elemental composition. Bulk hardness measurements were taken using Rockwell Hardness tester Model A1-BAS Serial No. 233 and values were recorded.

Observation

The results of chemical analysis & hardness survey (Table. 1) were found to conform to specification requirements. Visual examination of the round Die face showed a 180° crack across the top face (Fig. 3) but no such crack was found on the opposite bottom face. In the case of the elliptical Die the 180° crack was noticed on the top face along the major axis of the ellipse with minimum Die wall thickness (Fig. 4) but no such crack was noticed on the opposite bottom face.

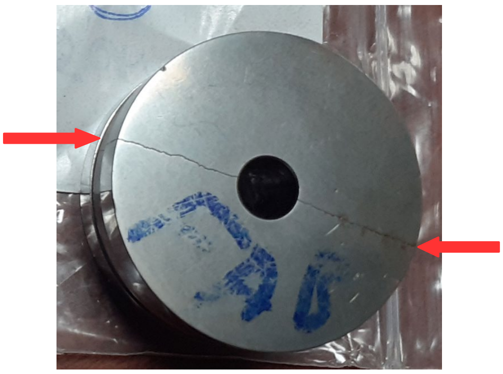

Fig. 3. Visual Examination -round Die

Fig. 4. Visual Examination – elliptical Die

Macro-examination (3-5X) revealed cracks of jagged morphology with rust marks on both the round & elliptical top Die faces (Fig. 5 & Fig.6) but sharp hairline cracks of 20 mm length connected to the those on the top faces were noticed on the outer diameter (OD) surfaces (Fig. 7 & Fig. 8). The macro-examination of the cut section of the round Die revealed jagged crack of length 19.5 mm on the Internal Diameter (ID) surface from the top face (Fig. 9) and cluster pit marks of 4 mm length around mid-height of the Die. The macroscopic observations of the elliptical Die were similar to that of the round Die (Fig, 10).

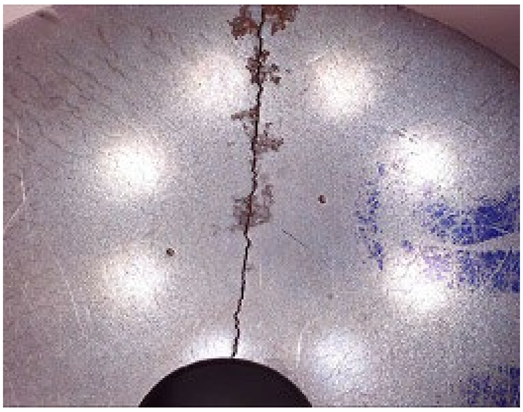

Fig.5. Macroexamination-round Die(rD) face

Fig. 6. Macroexamination-elliptical Die(eD) face

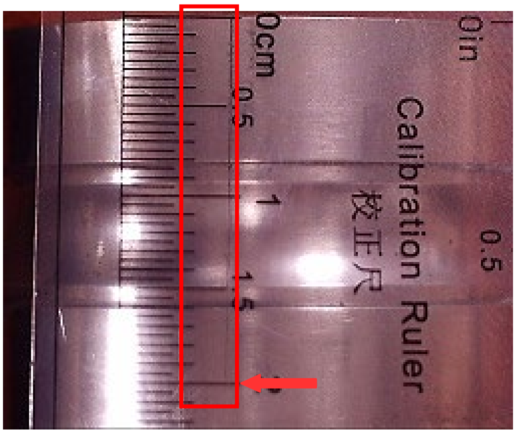

Fig.7. Macroexamination - rD OD surface

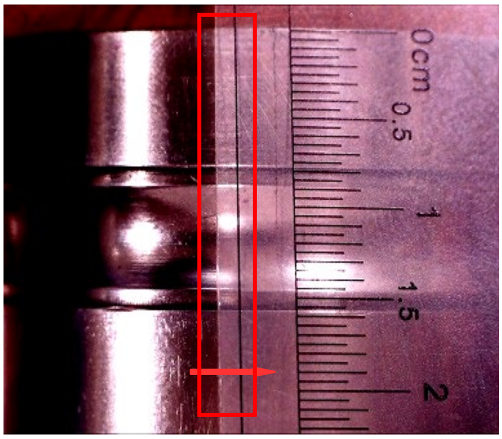

Fig. 8. Macroexamination – eD OD surface

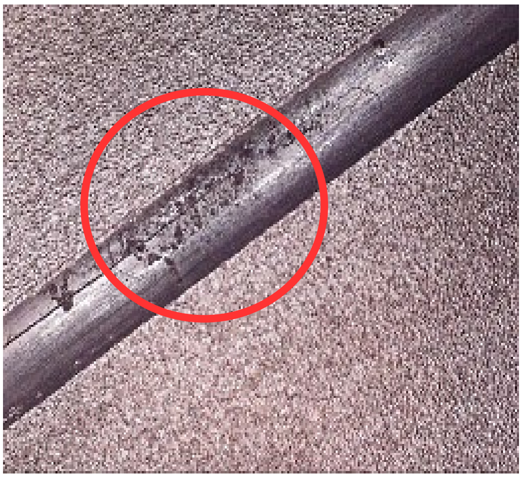

Fig. 9. Macroexamination - rD cluster pitmark

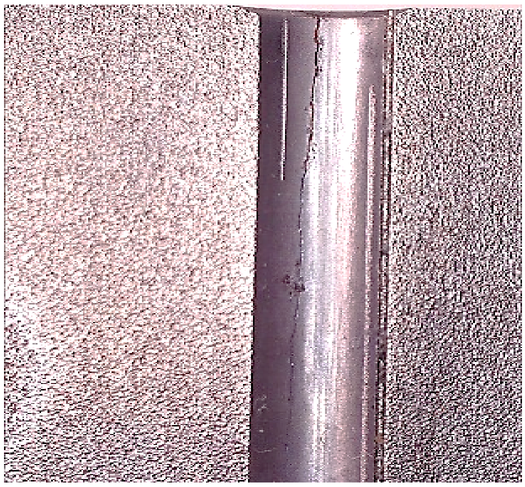

Fig.10. Macroexamination – eD cluster pitmark

Liquid Penetrant examination on the cut sections of both the round & elliptical Dies showed excessive bleeding around mid-height on the ID surface. Also, while it revealed linear indications from mid-height to the top face along the ID surface and on the top Die faces, no such indications were noticed near the bottom face (Fig. 11 & Fig. 12). Visual examination of the fractured surfaces of the round & elliptical Dies showed evidence of corrosion (Fig. 13 & Fig. 14) and clearly revealed the progression of corrosion into the material from the pitmarks observed on the ID surface.

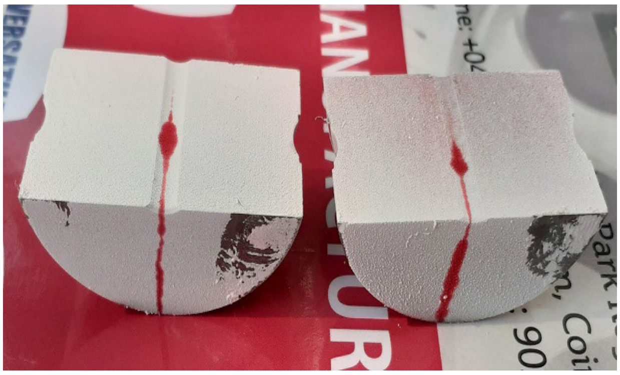

Fig. 11. Dye Penetrant Test – Round Die

Fig. 12. Dye Penetrant Test – Elliptical Die

Fig. 13. Fractured surface – Round Die

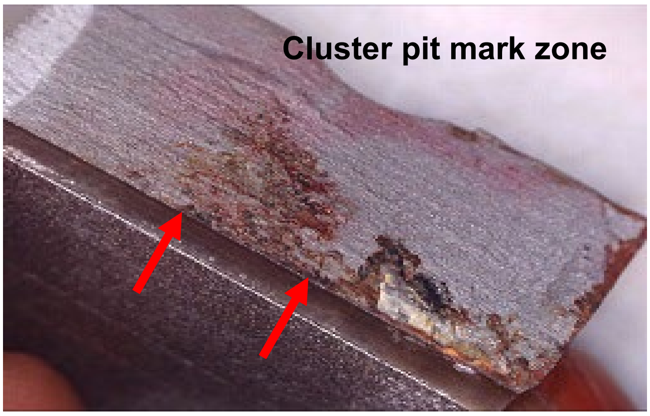

Fig.14. Fractured surface – Elliptical Die

Microstructural examination on samples taken on the top face of the round Die showed presence of 50% needle shaped carbides randomly distributed in tempered martensite matrix (Fig. 15). Also, several micro-cracks have been observed in the vicinity of the large crack on the top face of the round Die (Fig. 16). However, the microstructural examination on samples on the ID surface in the vicinity of the crack revealed severely banded carbides oriented along the axis of the Die (Fig. 17). The fractrography of sample taken at the cluster pit mark zone of the round Die revealed several micro-cracks originating from the ID surface propagating into the cross section of the Die (Fig. 18).

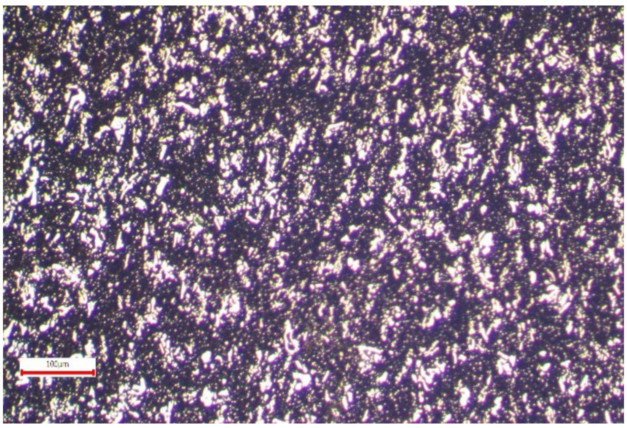

Fig. 15. Microstructure1 – Round Die face

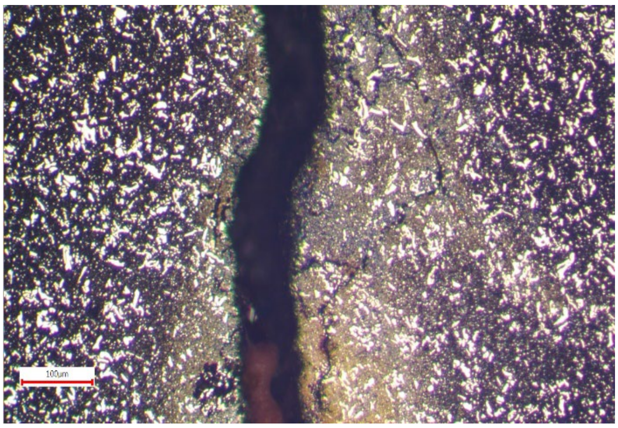

Fig. 16. Microstructure2 – Round Die face

Fig. 17. Microstructure – Banded structure

Fig. 18. Fractography – Clustered pit mark zone

While Scanning Electron Microscopy in the cluster pit mark zone of the round Die (Fig. 19) revealed the presence of multiple micro-cracks, SEM – EDX Analysis (Fig. 20) in the marked location close to the ID surface confirmed high content of FeO (81.77%) and low content of Cr_2O_3 (6.61%).

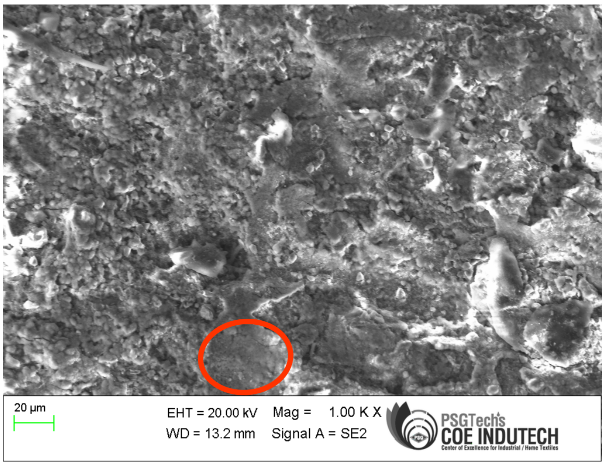

Fig. 19. SEM Analysis – Cluster pit mark zone

Spectrum: CORROSION.spx

Element	unn. C [wt.%]	norm. C [wt.%]	Atom. C [at.%]	Compound	norm. Comp. C [wt.%]	Error (3 Sigma) [wt.%]
Oxygen	19.37	25.29	51.93		0.00	7.52
Aluminium	2.28	2.97	3.62	Al2O3	5.61	0.43
Silicon	0.86	1.13	1.32	SiO2	2.41	0.20
Chromium	3.47	4.53	2.86	Cr2O3	6.61	0.38
Iron	48.67	63.56	37.39	FeO	81.77	3.99
Chlorine	0.37	0.48	0.45		0.48	0.13
Sulfur	0.10	0.13	0.14	SO3	0.33	0.10
Potassium	0.10	0.13	0.11	K2O	0.16	0.10
Calcium	0.31	0.41	0.33	CaO	0.57	0.12
Magnesium	0.45	0.58	0.79	MgO	0.96	0.18
Sodium	0.55	0.72	1.03	Na2O	0.97	0.23
Titanium	0.05	0.06	0.04	TiO2	0.11	0.09
Total:	76.58	100.00	100.00			

Fig. 20. SEM – EDX Analysis

Results & Discussion

The compression stage of the Tableting Process involves bringing together both the upper & lower punches under high hydraulic pressure through application of vertical load [75 – 80 KN] resulting in compaction of the formulation material by almost 80% to form the tablet [Fig.1]. This periodic application of load in the compression stage results in creating cyclic tensile hoop stress on the ID surface at the zone of compaction.

Owing to cyclic tensile hoop stresses generated in the Die cavity as mentioned above, the most prone region for the crack to initiate would be the zone of compaction on the ID surface for both the round & elliptical Dies. The cluster pit marks observed during macro-examination of the ID surface [Fig. 9] and the excessive bleeding noticed during Dye Penetrant examination of the ID surface [Fig. 11] at mid-height region of the Dies, confirm presence of multiple discontinuities in the said region and indicate the possibility of multiple crack initiation sites.

Since the ID surfaces of both the round & elliptical Dies had smooth machined finish, geometrical factors such as sharp corners etc. had played no role in crack initiation. Also, as the inclusion contents were very low (Table 2) and no apparent grain boundary carbides were noticed (Fig. 15), these microstructural features do not seem to have contributed towards crack initiation either. However, the surfaces obtained by fracturing the Dies along the crack (Fig. 13 & Fig. 14) showed moderate to high corrosion suggesting contribution of environmental factors for the crack initiation on the ID surface.

While the presence of corrosion on the fractured surfaces (Fig. 13 & Fig. 14) indicate that some of the ingredients in the formulation had been corrosive to AISI D3 Dies, the SEM-EDX analysis (Table.3) showed low percentage of Cr_2O_3 (6.61%) on the fractured surfaces in the cluster pit mark zone, despite the AISI D3 material having close to 12% of Chromium. This clearly indicates depletion of available Chromium towards preferential formation of Chromium Carbides, thereby making the material susceptible to corrosion due to insufficient Cr_2O_3 content. It can be inferred that the pit marks have been formed due to the ingredients used in tablet formulation that have been corrosive to the AISI D3 Dies getting smeared onto the ID surface at high pressure similar to that of a high pressure jet during every downward movement of the top punch in the compression stage of the Tableting process.

The microstructural analysis on sample taken on the ID surface of the round Die (Fig. 17) showed evidence of crack propagation direction along the Die axis. Presence of carbides preferentially oriented axially, known as banded carbides, could be observed. A banded structure can be defined as a segregated structure of approximately parallel bands of two different phases aligned in the working direction [1,2,6]. One of the major metallurgical problems reported in the literature for high carbon steels having Chromium greater than 11% is carbide banding [1]. The deleterious effects of carbide banding include reduced wear resistance, cracking along the carbides and reduction in fatigue resistance [1,5]. Parkar et. al. [3] reported that fatigue life of rolling element made from AMS 5749 [C ~ 1.1%; Cr ~ 14%; Mo ~ 4%] material reduced by four times due to carbide banding. Hence, the role of banded carbides found in the round Die along the axis, which happens to be the rolling direction, and their influence in the direction of crack propagation in the axial direction cannot be ruled out. The fractography at the cluster pit mark zone of the cut open / fractured surface (Fig 18) revealed multiple micro-cracks originating from the ID surface and propagating into the cross section of Die. Hence, it can be inferred that propagation of crack in the Die had been both in axial and transverse directions.

While it is clear from the evidence stated and the above discussion that crack origination had occurred on the ID surface in the cluster pit mark zone (Fig. 18) of the round Die, the jagged appearance of the crack of about 20 mm along the ID surface (Fig.5) and on the Die face suggests progressive nature of crack propagation. In addition, the appearance of hairline crack on the OD surfaces (Fig. 7) indicate accelerated crack propagation resulting in abrupt failure. The three phases of a typical fatigue failure [4,7] namely crack initiation, crack propagation through steady state & unsteady state and final fracture have been clearly evidenced in the Tableting Die failure.

Mechanism of Failure

The tableting process, which involves high load compaction reduces the height of formulation product in the Dies by almost 80% [Fig 1], thereby squeezing out the ingredients [water + other] and smearing them at high tensile hoop stress on the ID surface causing the formation of several minute corrosion pits in the mid-section of the Dies as the material has had insufficient corrosion resistance.

The cyclic loading [0-75 KN] applied during compaction had enabled formation of micro-cracks from the corrosion pits [8], which had acted as stress raisers. Multiple such micro-cracks so formed have had their origins in the mid-height region of the Dies. These micro-cracks had propagated both in the transverse direction i.e., perpendicular to the axis into the cross section of the Die [Fig 18] and in the axial direction on the ID surface [Fig. 17] through the joining of multiple micro-cracks to form a linear discontinuity, which steadily progressed towards the top & bottom faces of the Dies.

Owing to high vertical load applied by the top punch onto the formulation material inside the Die at a set frequency (1-3 cycles /sec), the tangential hoop stress experienced by the top half of the Die cavity is generally higher than that experienced by the bottom half of the Die cavity resulting in more intense crack propagation towards the top face of the Die compared to that of the bottom face. Also, the cyclic stresses experienced in the bottom half of the Die cavity were probably not sufficient to enable crack propogation along the ID surface till the bottom face of Die. With crack propagtion along the ID surface not reaching the bottom face of the Die, no crack propagation occurred on the bottom faces of both the round and elliptical Dies.

During the initial phase, the micro-cracks had propagated in a stable mode due to application of continuous cyclic stresses during tableting process in a corrosive environment. The jagged morphology of the crack along the ID surface and on the top face clearly indicate the evidence of progressive crack propagation in a stable mode. Though in the next phase, the cracks had accelerated over time, the propagation continued to be stable. Once the cracks originating from the ID surface progressively reached the outer edges of the Die face and those propagating into the cross section of the Die reached a critical size, both the round & elliptical Dies were unable to withstand the prevailing cyclic tensile hoop stresses experienced during operation leading to unstable mode of crack propagation followed by sudden fracture [4,7].

Conclusion

Cyclic stresses experienced during the compression stage of the Tableting Process together with the strong evidence of corrosive environment in service confirm that Corrosion Fatigue has been the predominant mode of failure for cracking of the Tableting Dies.

Recommendation

Following is the recommendation to prevent Die failures in service both in round & elliptical Dies:

1) Deployment of Martensitic Stainless Steel Dies with a Cr content of 16-18% with appropriate hardness in place of AISI D3 Dies.

2) Elimination of banded carbides observed in the microstructure through appropriate metal forming operation of Martensitic Stainless Steel material to avoid prefential orientation of grains and achieve near isotropic structure.

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