Delivering the Right Results

Predicting the Initial Deformation and Final Temperatures of Coal Ash Fusibility using the Measured Softening and Hemispherical Temperature

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INTRODUCTION

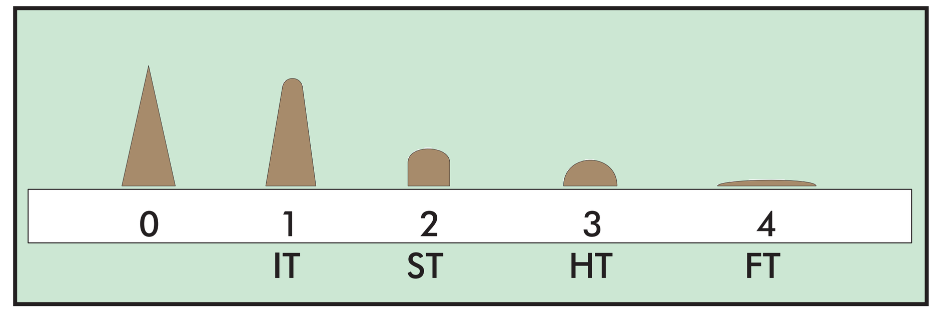
Ash fusion temperatures give an indication of the softening and melting behavior of fuel ash. Ash fusion temperatures are useful in predicting the slagging and fouling characteristics of fuel ash. The ASTM International standard test method for determining ash fusion temperatures (ASTM D 1857) uses four temperatures to characterize the fusion behavior of coal and coke ashes. The four transition temperatures are as follows:

Initial deformation temperature (IT) Temperature at which the first rounding of the apex of the cone occurs.

Softening temperature (ST) Temperature at which the cone has fused down to a spherical lump in which the height is equal to the width of the base.

Hemispherical temperature (HT) Temperature at which the cone has fused down to a hemispherical lump at which point the height is one-half the width of the base.

Fluid temperature (FT) Temperature at which the fused mass has spread out in a nearly flat layer with a maximum height of 1/16 in.



The temperature measurements are made by observing the behavior of ¼ x ¾ in. ASTM cones or DIN pins prepared from ash and mounted on a ceramic wafer. The observations may be made under either oxidizing (air) or reducing (60% CO and 40 % CO_2) atmosphere. The samples are heated rapidly to 800°C and then, at a rate of 8°C per minute (± 3°), to a maximum temperature (typically 1550°C). The four temperatures are assigned according to the definitions.

The AF700 Ash Fusion Determinator software automatically determines ash fusibility temperatures using Image Recognition Functions (IRF) within the software. Difficulties in determining IT and FT led to the investigation of using the measurable ST and HT values to determine IT and FT. A patent-pending series of equations was developed and verified with ash fusion temperatures from coal and coke ashes from widely varying sources on ASTM cones, DIN pins, and ASTM cones with broken tips.

METHODOLOGY

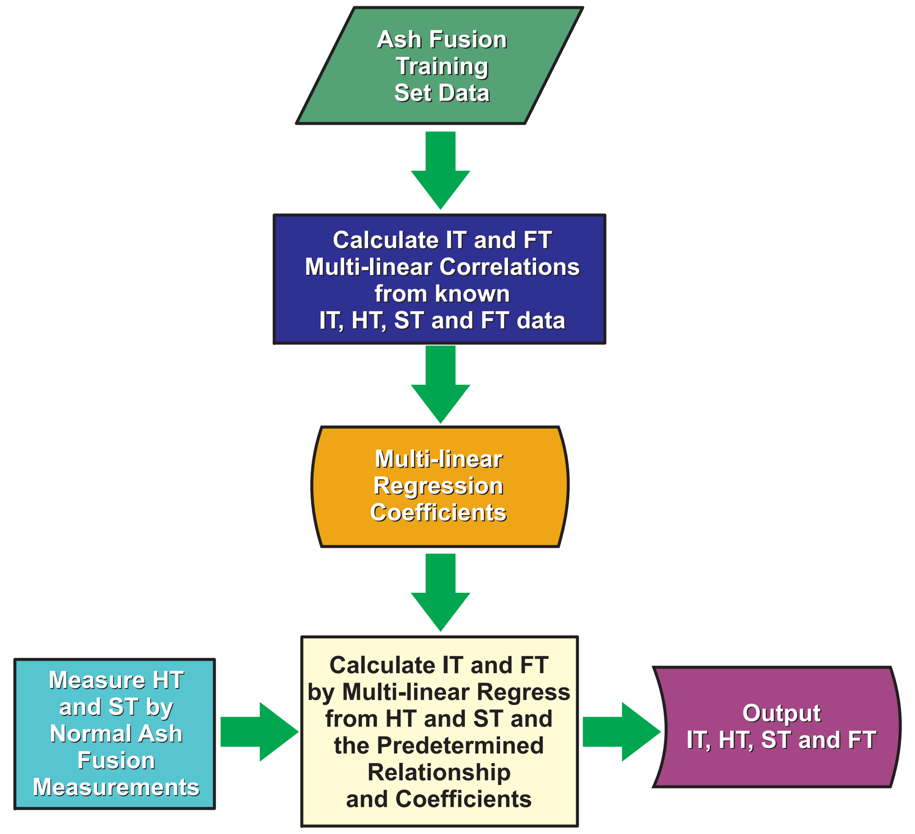
Equipment Required

- LECO AF700 Ash Fusion Determinator
- ASTM Cone mold or DIN pin mold

Procedure

1. Prepare either ASTM cone or DIN pin for analysis.
2. Place wafer with samples into AF700.
3. Analyze.

Predictive Algorithm Development Procedure



Typical ash fusion determination requires a well defined ASTM cone for accurate IT measurements. (see Figure 1)

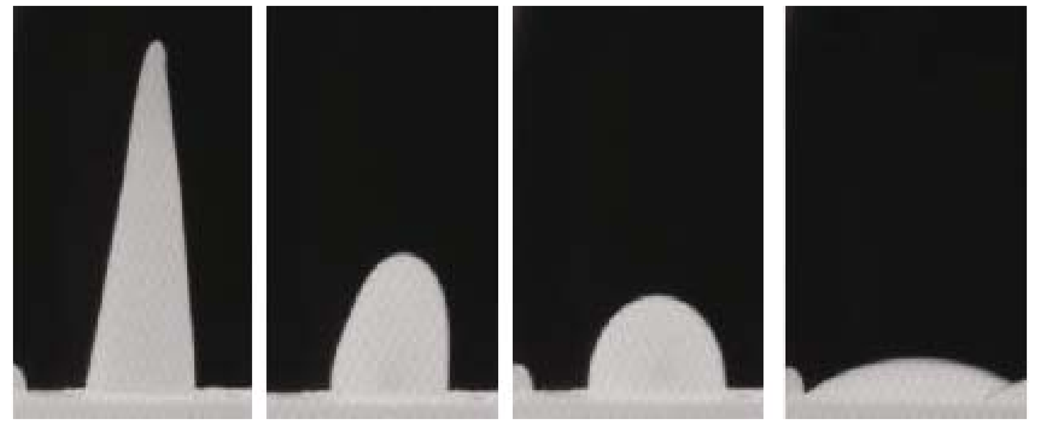


Figure 1. Sample 2000-1 ASTM cone

However, using the patent pending method, both DIN pins (Figure 2) and ASTM cones with broken tips (Figure 3) may be used to determine ash fusion values.

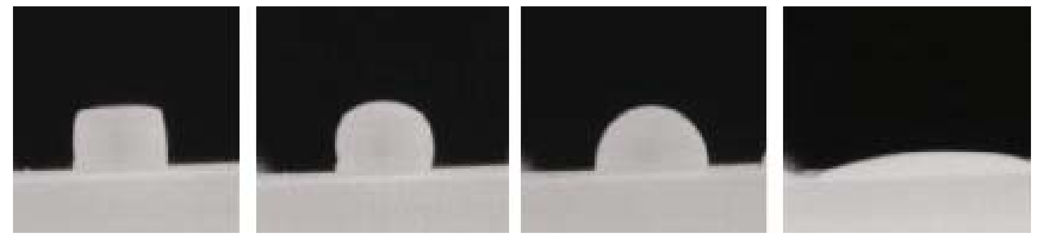


Figure 2. Sample 2000-1 DIN pin

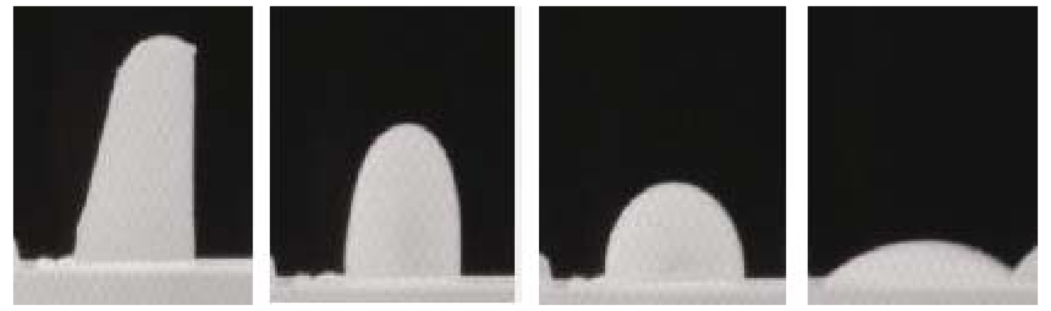


Figure 3. Sample 2000-1 ASTM cone with broken tip

Table 1. Measured vs. Calculated for Various Cone Shapes

Sample	IT ASTM	IT Predicted	ST	HT	FT ASTM	FT Predicted
2000-1 broken cone		1136	1156	1180	1270	1231
2000-1 pin	1072	1129	1151	1179	1284	1235
2000-1 cone	1077	1102	1136	1184	1272	1259
2000-1 pin		1132	1152	1176	1287	1230
2000-1 cone	1072	1127	1150	1179	1263	1236
average	1073	1125	1149	1180	1275	1238
Riley	1104		1147	1175	1225	
Canspex	1096	1117	1138	1165	1223	1222
std	2.4	13.4	7.6	2.9	10.0	11.9
rsd	0.22	1.19	0.66	0.24	0.79	0.96

RESULTS

Table 2. CANSPEX Data With Associated Manually Calculated Predicted Values

			IT		FT	
Sample	Measurement #	Labs	Consensus	Predicted	Consensus	Predicted
2000-1*	16	28	1096	1117	1223	1222
2001-1*	16	26	1104	1104	1193	1195
2001-2*	16	36	1216	1208	1333	1317
2001-3*	16	22	1111	1107	1256	1260
2001-4*	16	24	1215	1213	1253	1269
2002-1*	16	21	1134	1130	1267	1277
2002-2*	16	23	1221	1214	1294	1280
2002-3*	16	24	1247	1236	1340	1322
2002-4*	16	21	1114	1110	1284	1322
2003-1*	16	22	1282	1269	1362	1342
2003-2*	16	20	1330	1315	1411	1391
2003-3*	16	21	1184	1205	1355	1350
2003-4*	16	19	1322	1313	1474	1476
2004-1*	16	21	1433	1432	1559	1553
2004-2*	16	19	1526	1487	1542	1544
2004-3*	16	22	1099	1110	1178	1182
2004-4*	16	22	1183	1176	1420	1376
2005-1*	16	19	1390	1392	1486	1488
2005-2*	16	24	1159	1157	1229	1237
2005-3*	16	22	1106	1109	1199	1200
2005-4*	16	24	1249	1241	1289	1302
2006-1*	16	24	1207	1207	1243	1260
2006-2*	16	22	1152	1158	1334	1324
2006-3*	16	23	1302	1292	1441	1437
2006-4*	16	19	1249	1234	1370	1339
2007-1*	16	29	1134	1139	1292	1294
2007-2*	16	29	1171	1176	1215	1233
2007-3*	16	28	1159	1165	1214	1236
2007-4*	16	27	1390	1378	1493	1496
2008-1*	16	30	1248	1236	1343	1327

*Reducing environment

			IT		FT	
Sample	Measurement #	Labs	Consensus	Predicted	Consensus	Predicted
2000-1*	20	27	1243	1246	1345	1357
2001-1**	20	24	1122	1116	1217	1230
2001-2**	20	35	1246	1235	1354	1348
2001-3**	20	19	1277	1282	1389	1400
2001-4**	20	20	1237	1237	1275	1284
2002-1**	20	21	1207	1205	1316	1323
2002-2**	20	22	1248	1239	1316	1316
2002-3**	20	22	1277	1263	1361	1379
2002-4**	20	22	1306	1304	1388	1391
2003-1**	20	20	1315	1303	1380	1369
2003-2**	20	18	1340	1330	1410	1402
2003-3**	20	18	1368	1364	1422	1425
2003-4**	20	21	1393	1389	1492	1503
2004-1**	20	20	1504	1495	1580	1564
2004-2**	20	15	1232	1170	1467	1510
2004-3**	20	22	1205	1205	1273	1278
2004-4**	20	21	1252	1246	1428	1440
2005-1**	20	17	1465	1476	1519	1516
2005-2**	20	23	1165	1169	1241	1240
2005-3**	20	21	1125	1120	1223	1230
2005-4**	20	23	1246	1241	1292	1300
2006-1**	20	20	1218	1219	1266	1269
2006-2**	20	23	1372	1368	1424	1426
2006-3**	20	22	1398	1398	1475	1475
2006-4**	20	21	1268	1254	1389	1364
2007-1**	20	28	1208	1217	1340	1336
2007-2**	20	26	1214	1212	1283	1276
2007-3**	20	26	1178	1178	1224	1234
2007-4**	20	20	1474	1471	1511	1515
2008-1**	20	27	1276	1270	1357	1344

**Oxidizing environment

Table 3. LECO Comparative Study Using AF700 Integrated Prediction Software*

	ST		HT		IT				FT			
	Reference Value (Riley)	AF700	Reference Value (Riley)	AF700	Reference Values		AF700 Predicted		Reference Values		AF700 Predicted	
Sample					Riley	CANSPEX	ASTM	New AF700	Riley	CANSPEX	ASTM	New AF700
1997-4	1291	1306	1339	1332	1207		1229	**1269**	1387		1415	**1374**
1998-1	1431	1434	1475	1458	1383		1362	**1383**	1510		1492	**1489**
1998-2	1164	1147	1175	1155	1163		1237	**1139**	1197		1236	**1194**
1998-3	1296	1316	1333	1375	1250		1248	**1256**	1362		1394	**1444**
1998-4	1230	1236	1245	1264	1214		1205	**1204**	1308		1387	**1314**
1999-1	1153	1148	1168	1161	1138		1134	**1136**	1213		1230	**1204**
1999-2	1284	1269	1329	1325	1243		1237	**1215**	1406		1445	**1421**
1999-3	1193	1170	1204	1176	1177		1161	**1160**	1244		1203	**1212**
1999-4	1166	1179	1215	1203	1170		1128	**1157**	1244		1245	**1252**
2000-1	1147	1149	1175	1180	1104	1096	1073	**1125**	1225	1223	1275	**1238**
2000-2	1431	1423	1461	1457	1377		1358	**1368**	1487		1489	**1497**
2000-3	1170	1207	1186	1235	1154		1137	**1179**	1218		1306	**1280**
2000-4	1282	1263	1308	1281	1262		1246	**1235**	1374		1398	**1320**
2001-1	1123	1117	1144	1134	1107	1104	1092	**1106**	1219	1193	1214	**1183**
2001-4	1224	1230	1234	1234	1208	1215	1222	**1215**	1255	1253	1251	**1263**
2002-1	1164	1168	1207	1218	1127	1134	1126	**1129**	1265	1316	1335	**1293**
2002-2	1230	1232	1245	1242	1214	1221	1216	**1213**	1308	1294	1336	**1276**
2002-3	1265	1264	1284	1302	1245	1247	1244	**1223**	1340	1340	1383	**1358**
2002-4	1165	1129	1230	1175	1121	1114	1101	**1096**	1286	1284	1360	**1250**
2003-2	1348	1345	1363	1359	1325	1330	1318	**1312**	1413	1411	1447	**1385**
2003-4	1373	1371	1415	1403	1327	1322	1335	**1321**	1469	1474	1471	**1446**
2004-2	no melt											
2004-3	1121	1115	1138	1134	1105	1099	1072	**1102**	1189	1178	1207	**1186**
2004-4	1234	1194	1301	1288	1179	1183	1144	**1123**	1419	1420	1489	**1398**
2005-1	1439	1420	1460	1445	1362	1390	1369	**1371**	1485	1486	1473	**1476**
2005-3	1133	1116	1155	1148	1109	1106	1102	**1095**	1221	1199	1224	**1211**
2005-4	1257	1255	1268	1262	1243	1249	1246	**1235**	1288	1289	1273	**1291**
2006-1	1222	1220	1232	1224	1203	1207	1216	**1206**	1249	1243	1244	**1254**
2006-2	1220	1139	1262	1210	1180	1152	1119	**1091**	1337	1334	1334	**1300**
2006-3	1340	1326	1386	1365	1296	1302	1267	**1277**	1441	1441	1445	**1417**

*Reducing environment

OBSERVATIONS and COMMENTS

As stated, the objective of this study was to confirm that the difficult to measure ash fusion values of IT and FT can be accurately predicted from the more easily measurable ST and HT values. ASTM requires a repeatability limit of 30°C between runs. This means that with 95% confidence, the same operator on the same apparatus should be able to have successive determinations that are within 30°C of one another.

From the data shown, all but three of the predicted IT and FT values are well within the 30°C margin of error. The only sample that was consistently outside of the 30°C margin was sample 2004-2 which failed to fall within the specifications for both oxidizing and reducing environments in both studies. Sample 2004-4 fell outside of the specification when analyzed under reducing conditions in the CANSPEX Data evaluation, but was within 30°C of the consensus value when reanalyzed during the LECO Comparative Study. An average absolute difference of 13°C between consensus CANSPEX values and the LECO Comparative Study using the determination values of the AF700 software was observed. The CANSPEX Data with manually determined values showed an absolute average difference of 12°C under reducing conditions and 9°C under oxidizing conditions.

Overall, the predictive algorithm has shown to be a highly accurate tool for determining IT and FT from ST and HT values. The most highly valuable and interesting aspects are the ability of the calculations to account for defects such as broken tips on ASTM cones and the ability to use DIN pins. Figures 1-3 show a standard "good" ASTM cone (Fig. 1), a DIN pin (Fig. 2), and an ASTM cone with a broken tip (Fig. 3). The associated data can be seen in Table 1. Under "normal" conditions, an ASTM cone with a broken tip would be discarded resulting in a loss of operator time, and sample material because IT could not be determined. However, there was no statistical difference for the calculated vs. CANSPEX values when using broken tips. Some operators also prefer to use DIN pins which are easier to make. The IT is very difficult to determine when using DIN pins, but the calculated values from ST and HT are easily determined with good accuracy.

The new predictive algorithm used in conjunction with the AF700 is well within ASTM guidelines for accuracy and precision when determining IT and FT. By automatically terminating the analysis after the last deformation point has been reached for all samples, the algorithm increases throughput while also saving money by extending furnace lifetime. A variety of sample shapes and sizes may also be used, allowing for greater operator freedom to choose either ASTM or DIN pins. Broken ASTM cones may also be used, saving valuable preparation time.

Acknowledgement – Data employed to validate the predictive methods was taken from the CANSPEX Proficiency Test Program operated by Quality Associates International Canada Ltd.