APPENDIX A

Raw Data

Result y_1

1)Diameter (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.140	20.080	20.090	20.103
$A_0B_0C_1D_1$	20.080	20.080	20.120	20.093
$A_0B_1C_0D_1$	20.090	20.140	20.080	20.103
$A_0B_1C_1D_0$	20.090	20.080	20.090	20.087
$A_1B_0C_0D_0$	20.090	20.070	20.140	20.100
$A_1B_0C_1D_1$	20.070	20.100	20.090	20.087
$A_1B_1C_0D_1$	20.090	20.090	20.160	20.113
$A_1B_1C_1D_0$	20.070	20.140	20.120	20.110

2)Height (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.410	4.410	4.420	4.413
$A_0B_0C_1D_1$	4.410	4.420	4.410	4.413
$A_0B_1C_0D_1$	4.510	4.540	4.480	4.510
$A_0B_1C_1D_0$	4.410	4.390	4.460	4.420
$A_1B_0C_0D_0$	4.310	4.260	4.360	4.310
$A_1B_0C_1D_1$	4.420	4.440	4.410	4.423
$A_1B_1C_0D_1$	4.420	4.320	4.310	4.350
$A_1B_1C_1D_0$	4.440	4.410	4.390	4.413

3)Weight (g) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.554	9.553	9.553	9.553
$A_0B_0C_1D_1$	9.538	9.539	9.538	9.538
$A_0B_1C_0D_1$	9.628	9.629	9.627	9.628
$A_0B_1C_1D_0$	9.597	9.591	9.592	9.593
$A_1B_0C_0D_0$	9.448	9.449	9.446	9.448
$A_1B_0C_1D_1$	9.546	9.546	9.542	9.545
$A_1B_1C_0D_1$	9.437	9.438	9.436	9.437
$A_1B_1C_1D_0$	9.683	9.691	9.689	9.688

4)Diameter (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.05	20.06	20.06	20.057
$A_0B_0C_1D_1$	20.16	20.10	20.09	20.117
$A_0B_1C_0D_1$	20.12	20.11	20.12	20.117
$A_0B_1C_1D_0$	20.09	20.10	20.12	20.103
$A_1B_0C_0D_0$	20.14	20.12	20.15	20.137
$A_1B_0C_1D_1$	20.19	20.13	20.14	20.153
$A_1B_1C_0D_1$	20.15	20.20	20.23	20.193
$A_1B_1C_1D_0$	20.14	20.13	20.21	20.160

5)Height (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.37	4.33	4.23	4.310
$A_0B_0C_1D_1$	4.32	4.28	4.34	4.313
$A_0B_1C_0D_1$	4.45	4.39	4.37	4.403
$A_0B_1C_1D_0$	4.33	4.46	4.39	4.393
$A_1B_0C_0D_0$	4.34	4.43	4.41	4.393
$A_1B_0C_1D_1$	4.37	4.39	4.32	4.360
$A_1B_1C_0D_1$	4.40	4.31	4.33	4.347
$A_1B_1C_1D_0$	4.45	4.46	4.36	4.423

6) Weight (g) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.552	9.552	9.551	9.552
$A_0B_0C_1D_1$	9.543	9.539	9.538	9.540
$A_0B_1C_0D_1$	9.626	9.627	9.627	9.627
$A_0B_1C_1D_0$	9.588	9.587	9.586	9.587
$A_1B_0C_0D_0$	9.448	9.446	9.450	9.448
$A_1B_0C_1D_1$	9.543	9.545	9.544	9.544
$A_1B_1C_0D_1$	9.434	9.435	9.447	9.439
$A_1B_1C_1D_0$	9.683	9.681	9.684	9.683

7)Density (g/cm³) - before sintering

Experiment condition	
$A_0B_0C_0D_0$	7.171
$A_0B_0C_1D_1$	7.167
$A_0B_1C_0D_1$	7.200
$A_0B_1C_1D_0$	7.016
$A_1B_0C_0D_0$	6.841
$A_1B_0C_1D_1$	7.130
$A_1B_1C_0D_1$	7.227
$A_1B_1C_1D_0$	7.223

8)Density (g/cm³) - after sintering

Experiment condition	A	B	A / (A-B))* ρw
$A_0B_0C_0D_0$	9.5534	8.2098	7.096
$A_0B_0C_1D_1$	9.5401	8.2102	7.159
$A_0B_1C_0D_1$	9.6309	8.2972	7.207
$A_0B_1C_1D_0$	9.5882	8.2747	7.285
$A_1B_0C_0D_0$	9.4553	8.0770	6.846
$A_1B_0C_1D_1$	9.5450	8.2077	7.123
$A_1B_1C_0D_1$	9.4367	8.1046	7.070
$A_1B_1C_1D_0$	9.6829	8.3417	7.205

9)% of open porosity - after sintering

Experiment condition	w	ws	wss	
$A_0B_0C_0D_0$	9.552	9.5786	8.2463	2.00%
$A_0B_0C_1D_1$	9.540	9.5552	8.2150	1.13%
$A_0B_1C_0D_1$	9.627	9.6255	8.2879	-0.11%
$A_0B_1C_1D_0$	9.587	9.5886	8.1798	0.11%
$A_1B_0C_0D_0$	9.448	9.4716	8.1416	1.77%
$A_1B_0C_1D_1$	9.544	9.5731	8.2307	2.17%
$A_1B_1C_0D_1$	9.439	9.4351	8.1237	-0.30%
$A_1B_1C_1D_0$	9.683	9.6846	8.3389	0.12%

10) Vickers Hardness Number – after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	21.5	20.9	21.7	21.367
$A_0B_0C_1D_1$	26.8	23.7	25.1	25.200
$A_0B_1C_0D_1$	23.8	21.5	20.6	21.967
$A_0B_1C_1D_0$	24.9	25.4	22.5	24.267
$A_1B_0C_0D_0$	23.7	25.4	25.6	24.900
$A_1B_0C_1D_1$	34.5	31.7	32.2	32.800
$A_1B_1C_0D_1$	30.9	22.5	25.4	26.267
$A_1B_1C_1D_0$	29.0	27.4	23.7	26.700

Result y_2

1)Diameter (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.11	20.11	20.10	20.107
$A_0B_0C_1D_1$	20.13	20.08	20.14	20.117
$A_0B_1C_0D_1$	20.09	20.14	20.12	20.117
$A_0B_1C_1D_0$	20.09	20.08	20.08	20.083
$A_1B_0C_0D_0$	20.14	20.13	20.14	20.137
$A_1B_0C_1D_1$	20.12	20.15	20.14	20.137
$A_1B_1C_0D_1$	20.14	20.18	20.14	20.153
$A_1B_1C_1D_0$	20.14	20.14	20.14	20.140

2)Height (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.44	4.34	4.43	4.403
$A_0B_0C_1D_1$	4.45	4.41	4.40	4.420
$A_0B_1C_0D_1$	4.38	4.29	4.19	4.287
$A_0B_1C_1D_0$	4.28	4.43	4.39	4.367
$A_1B_0C_0D_0$	4.38	4.35	4.41	4.380
$A_1B_0C_1D_1$	4.34	4.40	4.27	4.337
$A_1B_1C_0D_1$	4.25	4.12	4.21	4.193
$A_1B_1C_1D_0$	4.27	4.35	4.33	4.317

3)Weight (g) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.583	9.584	9.583	9.583
$A_0B_0C_1D_1$	9.596	9.596	9.595	9.596
$A_0B_1C_0D_1$	9.580	9.582	9.580	9.581
$A_0B_1C_1D_0$	9.697	9.695	9.694	9.695
$A_1B_0C_0D_0$	9.587	9.589	9.588	9.588
$A_1B_0C_1D_1$	9.469	9.466	9.472	9.469
$A_1B_1C_0D_1$	9.422	9.422	9.422	9.422
$A_1B_1C_1D_0$	9.558	9.561	9.571	9.563

4)Diameter (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.11	20.11	20.10	20.107
$A_0B_0C_1D_1$	20.11	20.08	20.09	20.093
$A_0B_1C_0D_1$	20.11	20.15	20.10	20.120
$A_0B_1C_1D_0$	20.09	20.10	20.08	20.090
$A_1B_0C_0D_0$	20.16	20.15	20.14	20.150
$A_1B_0C_1D_1$	20.17	20.19	20.23	20.197
$A_1B_1C_0D_1$	20.20	20.13	20.15	20.160
$A_1B_1C_1D_0$	20.13	20.14	20.15	20.140

5)Height (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.29	4.32	4.34	4.317
$A_0B_0C_1D_1$	4.31	4.29	4.33	4.310
$A_0B_1C_0D_1$	4.41	4.21	4.28	4.300
$A_0B_1C_1D_0$	4.36	4.40	4.26	4.340
$A_1B_0C_0D_0$	4.32	4.27	4.26	4.283
$A_1B_0C_1D_1$	4.37	4.33	4.25	4.317
$A_1B_1C_0D_1$	4.31	4.26	4.35	4.307
$A_1B_1C_1D_0$	4.24	4.29	4.34	4.290

6) Weight (g) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.582	9.583	9.579	9.581
$A_0B_0C_1D_1$	9.593	9.596	9.597	9.595
$A_0B_1C_0D_1$	9.581	9.580	9.582	9.581
$A_0B_1C_1D_0$	9.694	9.693	9.694	9.694
$A_1B_0C_0D_0$	9.584	9.587	9.582	9.584
$A_1B_0C_1D_1$	9.472	9.471	9.471	9.471
$A_1B_1C_0D_1$	9.420	9.425	9.422	9.422
$A_1B_1C_1D_0$	9.556	9.555	9.554	9.555

7)Density (g/cm^3) - before sintering

Experiment condition	
$A_0B_0C_0D_0$	7.126
$A_0B_0C_1D_1$	7.144
$A_0B_1C_0D_1$	7.251
$A_0B_1C_1D_0$	7.261
$A_1B_0C_0D_0$	7.089
$A_1B_0C_1D_1$	7.175
$A_1B_1C_0D_1$	7.248
$A_1B_1C_1D_0$	7.267

8)Density (g/cm^3) - after sintering

Experiment condition	A	B	A / (A-B))* ρw
$A_0B_0C_0D_0$	9.5868	8.2428	7.119
$A_0B_0C_1D_1$	9.5961	7.8992	5.644
$A_0B_1C_0D_1$	9.5819	8.227	7.058
$A_0B_1C_1D_0$	9.6987	8.2544	6.702
$A_1B_0C_0D_0$	9.5913	8.2406	7.087
$A_1B_0C_1D_1$	9.4711	8.1366	7.083
$A_1B_1C_0D_1$	9.4214	8.0717	6.966
$A_1B_1C_1D_0$	9.5577	8.2363	7.219

9)% of open porosity - after sintering

Experiment condition	w	ws	wss	
$A_0B_0C_0D_0$	9.581	9.6053	8.2707	1.82%
$A_0B_0C_1D_1$	9.595	9.6172	8.1233	1.49%
$A_0B_1C_0D_1$	9.581	9.5801	8.138	-0.06%
$A_0B_1C_1D_0$	9.694	9.6925	7.9858	-0.09%
$A_1B_0C_0D_0$	9.584	9.6132	8.2652	2.17%
$A_1B_0C_1D_1$	9.471	9.4929	8.1533	1.63%
$A_1B_1C_0D_1$	9.422	9.4252	7.9006	0.21%
$A_1B_1C_1D_0$	9.555	9.5569	8.2383	0.14%

10) Vickers Hardness Number – after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	22.70	21.2	21.0	21.633
$A_0B_0C_1D_1$	22.8	25.2	23.2	23.733
$A_0B_1C_0D_1$	19.8	22.5	24.0	22.100
$A_0B_1C_1D_0$	21.5	22.7	24.0	22.733
$A_1B_0C_0D_0$	22.2	23.8	22.2	22.733
$A_1B_0C_1D_1$	31.1	32.2	33.0	32.100
$A_1B_1C_0D_1$	27.0	27.2	26.0	26.733
$A_1B_1C_1D_0$	25.8	26.8	27.9	26.833

Result y_3

1)Diameter (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.12	20.13	20.17	20.140
$A_0B_0C_1D_1$	20.13	20.12	20.13	20.127
$A_0B_1C_0D_1$	20.11	20.13	20.12	20.120
$A_0B_1C_1D_0$	20.16	20.13	20.14	20.143
$A_1B_0C_0D_0$	20.12	20.14	20.11	20.123
$A_1B_0C_1D_1$	20.15	20.12	20.13	20.133
$A_1B_1C_0D_1$	20.12	20.13	20.11	20.120
$A_1B_1C_1D_0$	20.13	20.19	20.13	20.150

2)Height (mm) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.40	4.47	4.42	4.430
$A_0B_0C_1D_1$	4.45	4.51	4.42	4.460
$A_0B_1C_0D_1$	4.22	4.36	4.34	4.307
$A_0B_1C_1D_0$	4.25	4.43	4.36	4.347
$A_1B_0C_0D_0$	4.41	4.49	4.36	4.420
$A_1B_0C_1D_1$	4.37	4.47	4.41	4.417
$A_1B_1C_0D_1$	4.43	4.29	4.32	4.347
$A_1B_1C_1D_0$	4.33	4.28	4.37	4.327

3)Weight (g) - Green compact

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.760	9.758	9.762	9.760
$A_0B_0C_1D_1$	9.774	9.775	9.777	9.775
$A_0B_1C_0D_1$	9.616	9.617	9.617	9.617
$A_0B_1C_1D_0$	9.665	9.666	9.666	9.666
$A_1B_0C_0D_0$	9.601	9.606	9.599	9.602
$A_1B_0C_1D_1$	9.706	9.703	9.709	9.706
$A_1B_1C_0D_1$	9.493	9.499	9.496	9.496
$A_1B_1C_1D_0$	9.672	9.676	9.681	9.676

4)Diameter (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	20.12	20.15	20.18	20.150
$A_0B_0C_1D_1$	20.10	20.09	20.11	20.100
$A_0B_1C_0D_1$	20.10	20.12	20.10	20.107
$A_0B_1C_1D_0$	20.09	20.11	20.13	20.110
$A_1B_0C_0D_0$	20.16	20.12	20.15	20.143
$A_1B_0C_1D_1$	20.17	20.16	20.20	20.177
$A_1B_1C_0D_1$	20.15	20.19	20.16	20.167
$A_1B_1C_1D_0$	20.17	20.18	20.15	20.167

5)Height (mm) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	4.51	4.53	4.40	4.480
$A_0B_0C_1D_1$	4.41	4.49	4.39	4.430
$A_0B_1C_0D_1$	4.36	4.23	4.33	4.307
$A_0B_1C_1D_0$	4.36	4.35	4.41	4.373
$A_1B_0C_0D_0$	4.36	4.38	4.24	4.327
$A_1B_0C_1D_1$	4.37	4.47	4.39	4.410
$A_1B_1C_0D_1$	4.28	4.35	4.29	4.307
$A_1B_1C_1D_0$	4.38	4.27	4.29	4.313

6) Weight (g) - after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	9.760	9.759	9.761	9.760
$A_0B_0C_1D_1$	9.778	9.775	9.776	9.776
$A_0B_1C_0D_1$	9.618	9.617	9.614	9.616
$A_0B_1C_1D_0$	9.664	9.672	9.666	9.667
$A_1B_0C_0D_0$	9.602	9.603	9.599	9.601
$A_1B_0C_1D_1$	9.714	9.711	9.710	9.712
$A_1B_1C_0D_1$	9.493	9.495	9.493	9.494
$A_1B_1C_1D_0$	9.671	9.672	9.673	9.672

7)Density (g/cm^3) - before sintering

Experiment condition	
$A_0B_0C_0D_0$	7.108
$A_0B_0C_1D_1$	6.704
$A_0B_1C_0D_1$	7.267
$A_0B_1C_1D_0$	7.254
$A_1B_0C_0D_0$	7.105
$A_1B_0C_1D_1$	7.098
$A_1B_1C_0D_1$	7.268
$A_1B_1C_1D_0$	7.252

8)Density (g/cm^3) - after sintering

Experiment condition	A	B	A / (A-B))* ρw
$A_0B_0C_0D_0$	9.7666	8.3753	7.006
$A_0B_0C_1D_1$	9.7787	8.3286	6.730
$A_0B_1C_0D_1$	9.6192	8.2987	7.270
$A_0B_1C_1D_0$	9.6665	8.2889	7.003
$A_1B_0C_0D_0$	9.6038	8.2528	7.094
$A_1B_0C_1D_1$	9.7136	8.3432	7.074
$A_1B_1C_0D_1$	9.4959	8.1755	7.177
$A_1B_1C_1D_0$	9.671	8.2367	6.729

9)% of open porosity - after sintering

Experiment condition	w	ws	wss	
$A_0B_0C_0D_0$	9.76	9.7886	8.1094	1.70%
$A_0B_0C_1D_1$	9.76	9.7995	8.3389	2.70%
$A_0B_1C_0D_1$	9.616	9.6169	8.2998	0.07%
$A_0B_1C_1D_0$	9.667	9.6662	8.3323	-0.06%
$A_1B_0C_0D_0$	9.601	9.6201	8.2726	1.42%
$A_1B_0C_1D_1$	9.712	9.7324	8.3575	1.48%
$A_1B_1C_0D_1$	9.494	9.4977	8.0557	0.26%
$A_1B_1C_1D_0$	9.672	9.6756	8.3389	0.27%

10) Vickers Hardness Number – after sintering

Experiment condition	1	2	3	Mean
$A_0B_0C_0D_0$	21.2	20.3	23.2	21.567
$A_0B_0C_1D_1$	22.5	25.6	24.2	24.100
$A_0B_1C_0D_1$	21.9	23.2	22.7	22.600
$A_0B_1C_1D_0$	21.6	24.5	25.1	23.733
$A_1B_0C_0D_0$	23.3	27.2	23.0	24.500
$A_1B_0C_1D_1$	31.1	33.9	36.0	33.667
$A_1B_1C_0D_1$	30.2	31.7	33.0	31.633
$A_1B_1C_1D_0$	26.2	28.3	25.4	26.633

APPENDIX B

Microwave Oven Technical Specification

Model	Samsung M197DN
Power Source	240V ~ 50Hz
Power Consumption	1400W
Microwave Output Power	100W / 1000W (IE C-705)
Operating Frequency	2450MHz
Magnetron	OM 75 P(31)
Cooling Method	Cooling fan motor
Dimensions (WxHxD) Outside	 517 x 297 x 420 mm
Oven Cavity	336 x 241 x 349 mm
Volume	28 liter
Weight Net	16.5 kg approximately

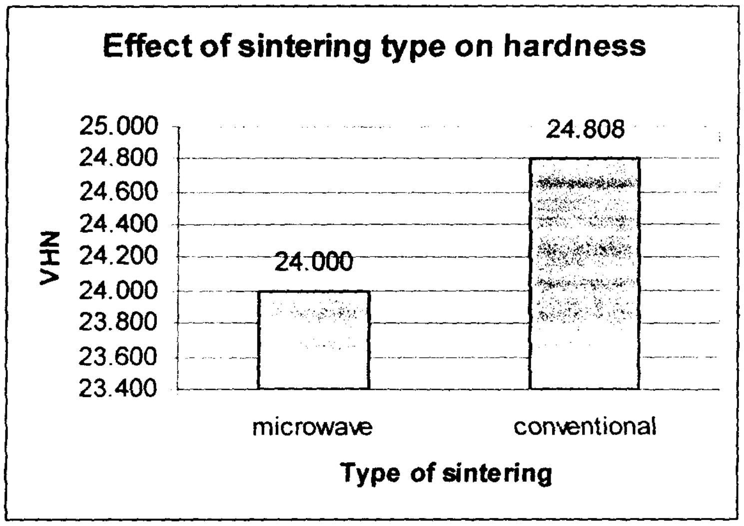


Figure 5.5.1.2 Effect sintering type on hardness

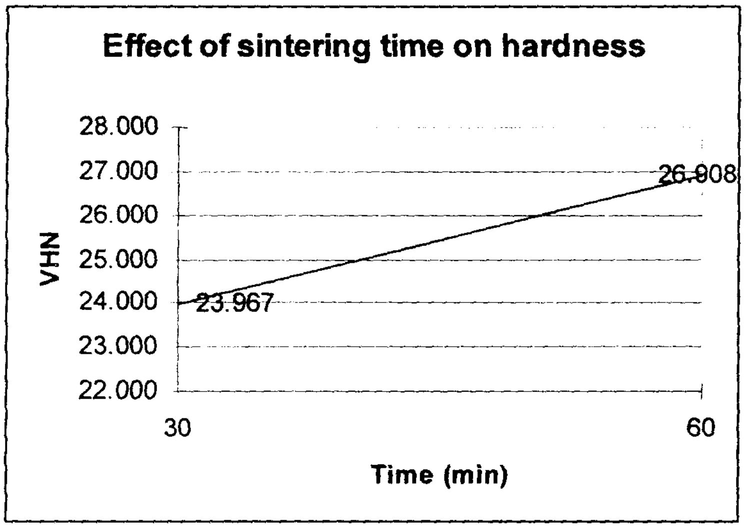


Figure 5.5.1.3 Effect of sintering time on hardness

5.5.2 Pareto Anova

The optimum level for each factor will be at which the average of the "iso-level" value of S/N ratio is maximum. The average iso-level value is determined from the values of S/N ratios at low and high levels of the factors. For example, the average S/N ratio for hardness at low level of factor A is determined from experimental runs 1,2,3 and 4 and for high level from experimental runs 5,6,7 and 8. Similarly, for other factors these iso-levels values are determined and recorded in Table 5.5.2. Figure 5.5.2 shows the iso-level SN values for hardness at different level of parameters.

Factors	Hardness at Low Level (0)	Hardness at High Level (1)	Difference
A Sintering temperature	27.188	28.846	1.658
B Compaction pressure	28.076	27.958	0.118
Interaction AxB	27.958	28.075	0.117
C Type of sintering	27.528	28.506	0.978
Interaction AxC	28.114	27.920	0.194
D Sintering time	27.559	28.475	0.916
Interaction AxD	28.336	27.698	0.638

Table 5.5.2 Average iso-level SN ratio values for hardness

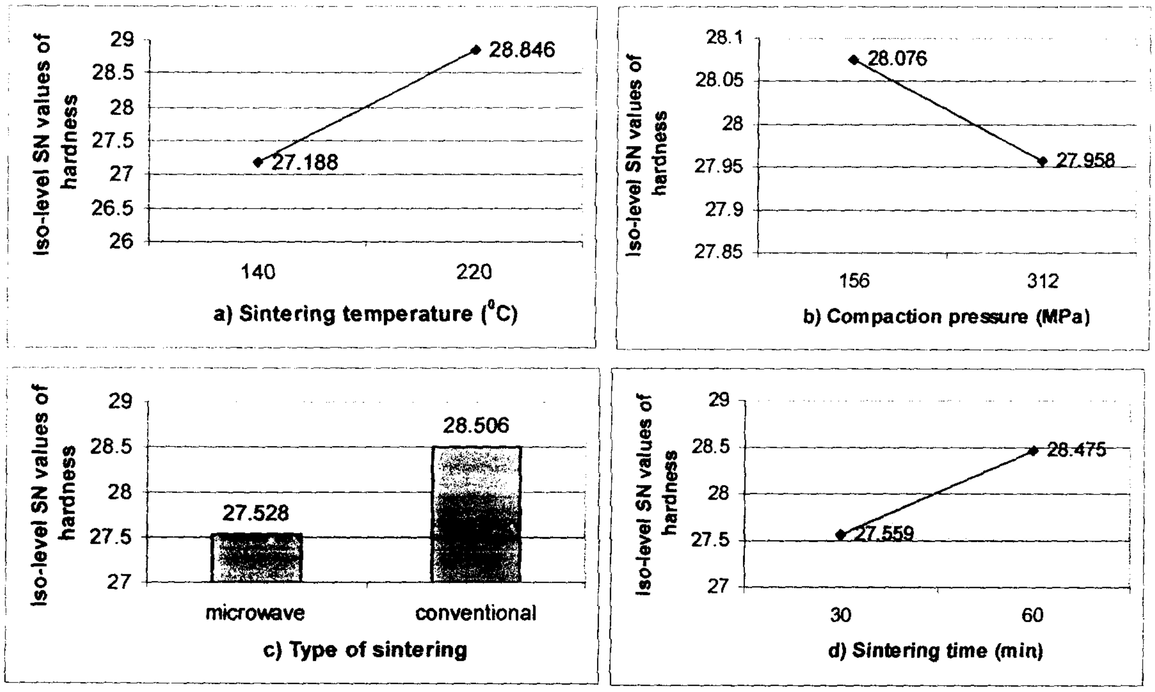


Figure 5.5.2 Iso-level SN values of hardness at different levels of parameter a)sintering temperature; b)compaction pressure; c)type of sintering; d)sintering time

From Table 5.5.2, one can see that the optimum combination of input parameters for highest hardness is $A_1B_0C_1D_1$ (220°C sintering temperature, 156MPa compaction pressure, conventionally sintered and 60 minutes sintering time). The Pareto Analysis of Variance (ANOVA) applied to this data gives the same optimum level of controllable factors as the iso-level technique and shown in Table 5.5.2.1

Table 5.5.2.1 Pareto Analysis of Variance (ANOVA) for hardness

Factor and interactions		A	B	AxB	C	AxC	D	AxD	Total
Sum at factor level	0	108.751	112.304	111.833	110.110	112.455	110.234	113.343	sum of 0,1 level = 224.13
	1	115.383	111.830	112.301	114.024	111.679	113.900	110.791	
Difference at 2 levels		6.632	0.474	0.468	3.914	0.776	3.666	2.552	
Square of difference		43.983	0.225	0.219	15.319	0.602	13.440	6.512	80.301
Contribution ratio (%)		54.77	0.28	0.27	19.08	0.75	16.74	8.11	100.00

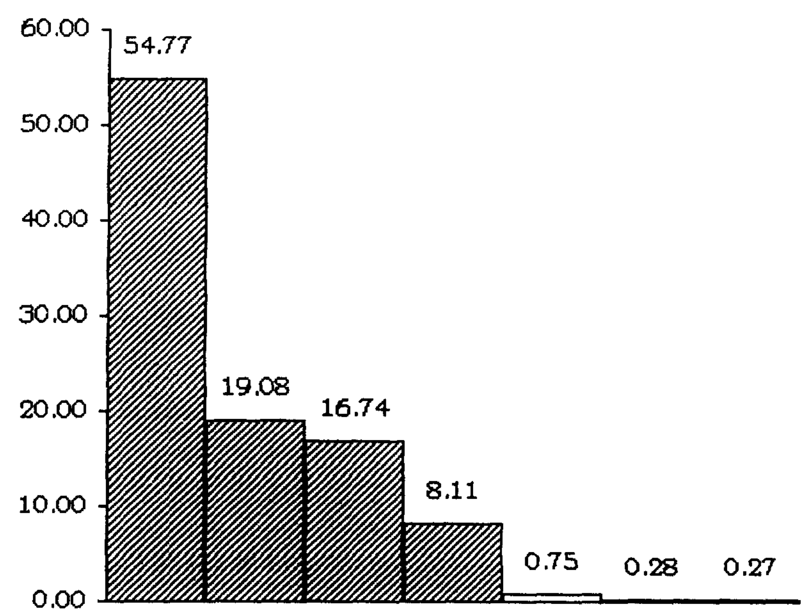


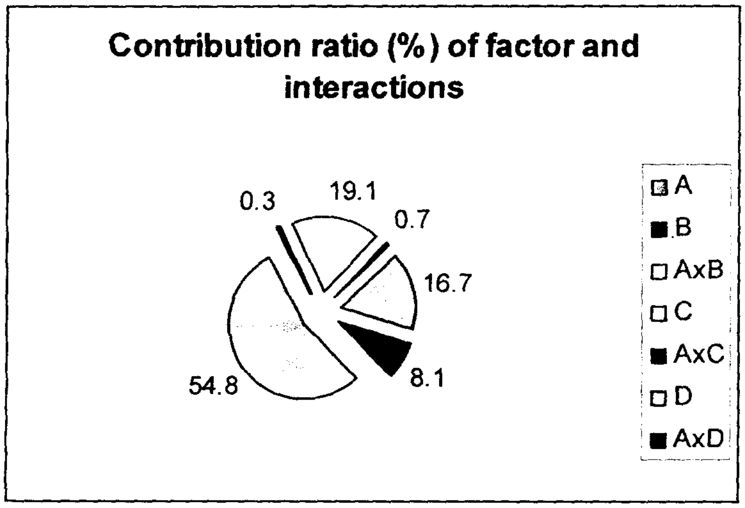
Figure 5.5.2.1 Pareto diagram for hardness

Factor and interactions	A	C	D	AxD	AxC	B	AxB

Table 5.5.2.1 continued

Cumulative contribution ratio (%)	54.77	73.85	90.59	98.70	99.45	99.73	100.00
Check the significant interactions		D_0		D_1			
	A_0	27.440+26.657=54.10		27.72+26.934=54.65		A_1D_1 is optimal	
	A_1	28.537+27.6 = 56.14		28.919+30.327 = 59.25			
Optimum combination of significant factor levels	$A_1C_1D_1$						
Remarks	The significant factors are chosen from the left hand side in the above Pareto diagram which cumulatively contribute about 98.7%						
Overall optimum conditions for all factors	A_1 = sintering temperature, 220°C B_0 = compaction pressure, 156MPa C_1 = conventional sintering D_1 = sintering time, 60min						
Estimate of error variance	Ve= (pooled variation of non-significat sources)/ (pooled degrees of freedom of non-significant sources) = 0.2247/8 = 0.0281						

From the above Pareto ANOVA (Table 5.5.2.1), it is seen that factor A, sintering temperature, contributes 54.8% to the hardness, factor C, type of sintering 19% and factor D, time of sintering 17 % as shown in Figure 5.5.2.2. The higher the sintering temperature and larger the sintering time, the better will be the sintering and hence higher will be the hardness and these factors also interact with each other. This study has shown that conventional sintering gives greater hardness. As has already been pointed out, the nature of heating in conventional sintering favors the better precipitation of harder phases which is evident from microstructure (Figure 5.6.5). In general, the precipitation of harder phases of copper-tin and antimony- tin intermetallic compounds is not good when microwave heating is performed as is evident from its microstructures in Figure 5.6, Figure 5.6.2, Figure 5.6.4 and Figure 5.6.6.



A= 54.77%, B = 0.28%, Ax B = 0.27%, C = 0.2%, Ax C = 0.9%, D = 0% and Ax D = 1.9%

Figure 5.5.2.2 Contribution ratio in percentage of factor and interactions

5.6 Microstructure

The microstructures of the specimens under each experimental condition were observed under optical metallurgical microscope with magnification X500. For magnification X500, 1 cm = 20µ.

In these microstructures, copper particles (reddish yellow color phase) and intermetallic compounds of Sn-Sb and Cu_6-Sn_5 (purple color phase) are embedded in the matrix of sintered tin, because sintering is performed at temperatures 140°C and 220°C (70 to 90% of the melting point of tin). These temperatures are much lower than the melting point of copper. At this temperature copper will not diffuse into tin to form an alloy. So the resultant structure shall be a composite rather than an alloy. Intermetallic compounds of copper and antimony appear as purple color phase in the structure. The pores appear as black spots.

In general it is observed that in microwave sintered specimens, the hard copper-tin and antimony-tin inter-metallic compound phase has not been fully formed (Figs 5.6, 5.6.2, 5.6.4 and 5.6.6). Therefore, the hardness values of the specimens sintered by microwave heating are in general lower the hardness values of specimens sintered under argon gas in a conventional heating furnace.

From these figures it is also observed that the open pores porosity is not much affected by type of sintering, even though microwave sintering gives slightly higher porosity as compared to conventional sintering. When microwave sintering is performed, the pores formed are of smaller size and larger in numbers as compared to the conventional sintering and have more rounded and smooth edges.

The microstructures of the specimens under each experimental condition are discussed below for the respective figures.

Figure 5.6 shows the microstructure of a specimen sintered with microwave heating, at temperature 140°C and compaction pressure 156 MPa for 30 minutes. The specimen under this condition has shrunk with a diameter change of 0.06%. It has 7.074 g/cm^3 density and the highest porosity of 1.84%. Its hardness is lowest at 21.52.The purple colored crystals of the inter-metallic compounds are not fully developed. Pores are of smaller size, overall porosity is low and the pores are rounded in shape.

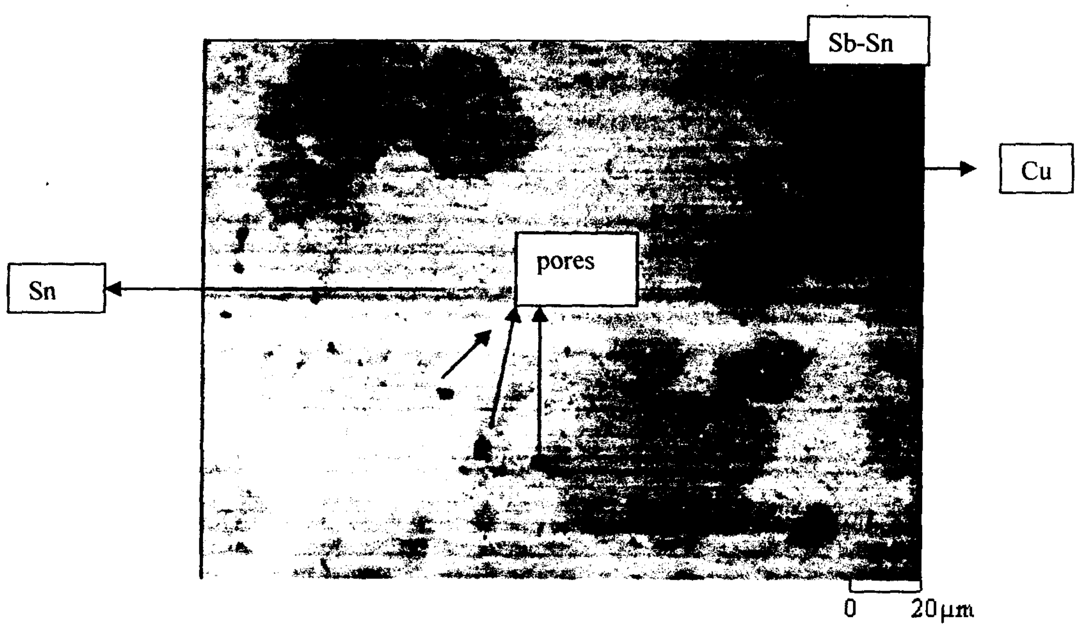


Figure 5.6 Microstructure of Experiment 1 (140°C, 156 MPa, microwave, 30 min)

Figure 5.6.1 shows the micro structure of specimens under similar compaction and sintering temperature as above, but sintered conventionally for a longer period (60minutes). The specimen under this condition has shrunk with a diameter change of 0.04%. It has the lowest density of 6.511 g/cm^3 and the porosity of 1.77%. Its hardness is 24.34.It may be observed that the inter-metallic phase crystals (purple colored islands) are fully formed, copper phase (reddish yellow color areas) reduced, showing that some of the copper has combined with tin to form hard inter-metallic compound phase. Pore size is large and of irregular shape.

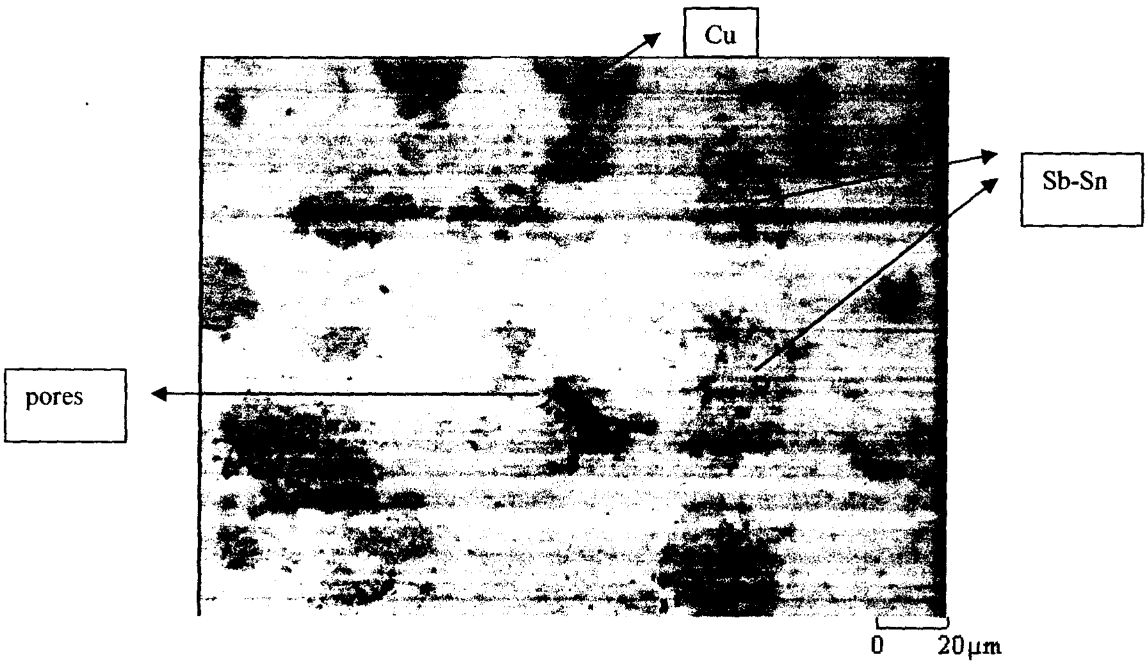


Figure 5.6.1 Microstructure of Experiment 2 (140°C, 156 MPa, Ar gas, 60 min)

The specimen shown in Figure 5.6.2, is microwave sintered at 140°C for 60 minutes and compacted at 312 MPa. The specimen under this condition has swelled with a diameter change of 0.01% which is the highest accuracy. It has the highest density of 7.178 g/cm^3 and the lowest porosity of 0.02%. Its hardness is 22.22.The comparison of Figure 5.6.2 and Figure 5.6.7 show that even on sintering the specimens compacted at higher pressure for a longer period of time, microwave heating has failed to segregate the hard crystal phases of inter-metallic compounds. Copper islands have larger area. The pores are elongated, but the edges are smooth and rounded. The sizes of pores are larger than for the specimens compacted at lower pressure and sintered for a shorter period.

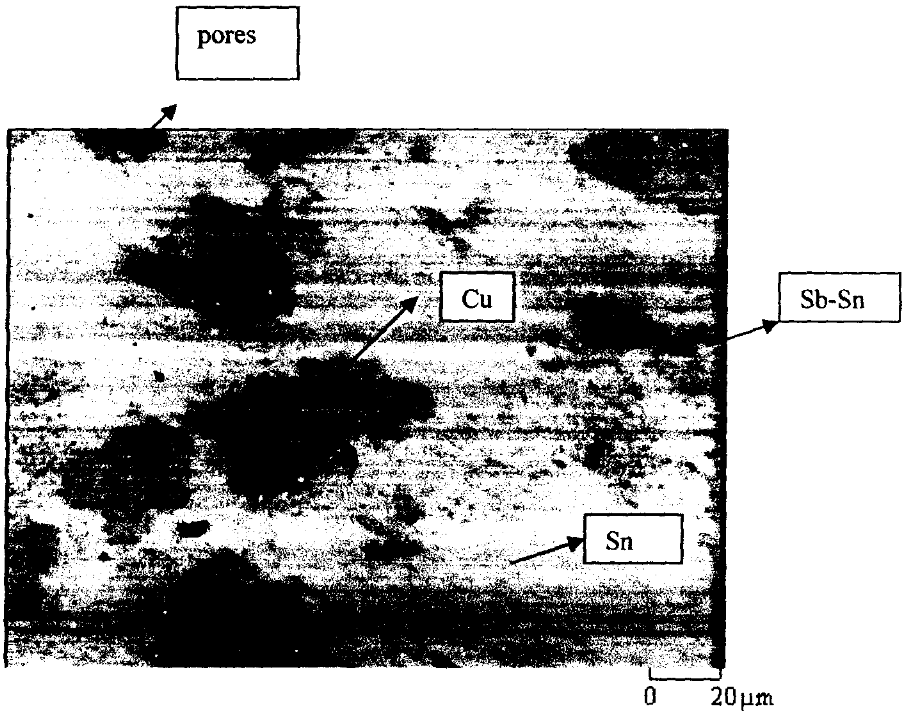


Figure 5.6.2 Microstructure of Experiment 3 (140°C, 312 MPa, microwave, 60 min)

Figure 5.6.3 shows that in the specimens compacted at higher pressure, but sintered at lower temperature for a shorter period, even under conventional sintering the hard inter-metallic phase do not fully segregate. The pores are more irregular in this case as compared to conventional sintering. The specimen under this condition has shrunk with a diameter change of 0.02%. It has 6.997 g/cm^3 density and the porosity of 0.04%. Its hardness is 23.58.

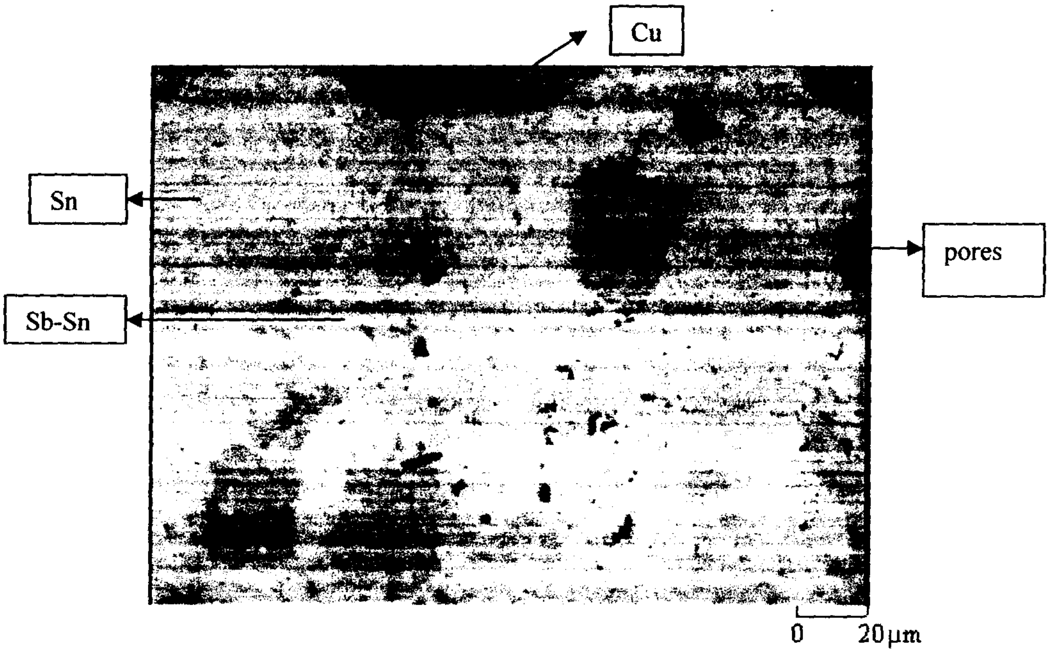


Figure 5.6.3 Microstructure of Experiment 4 (140°C, 312 MPa, Ar gas, 30 min)

The specimen shown in Figure 5.6.4, is sintered in a microwave furnace at 220°C for 30 minutes and compacted at 156 MPa. The specimen under this condition has swelled with a diameter change of 0.12%. It has 7.009 g/cm^3 density and porosity of 1.79%. Its hardness is 24.04. Fig 5.6.4 shows that high temperature sintering favours the formation of some hard inter-metallic phases even with microwave heating and low compaction pressure and smaller time of sintering.

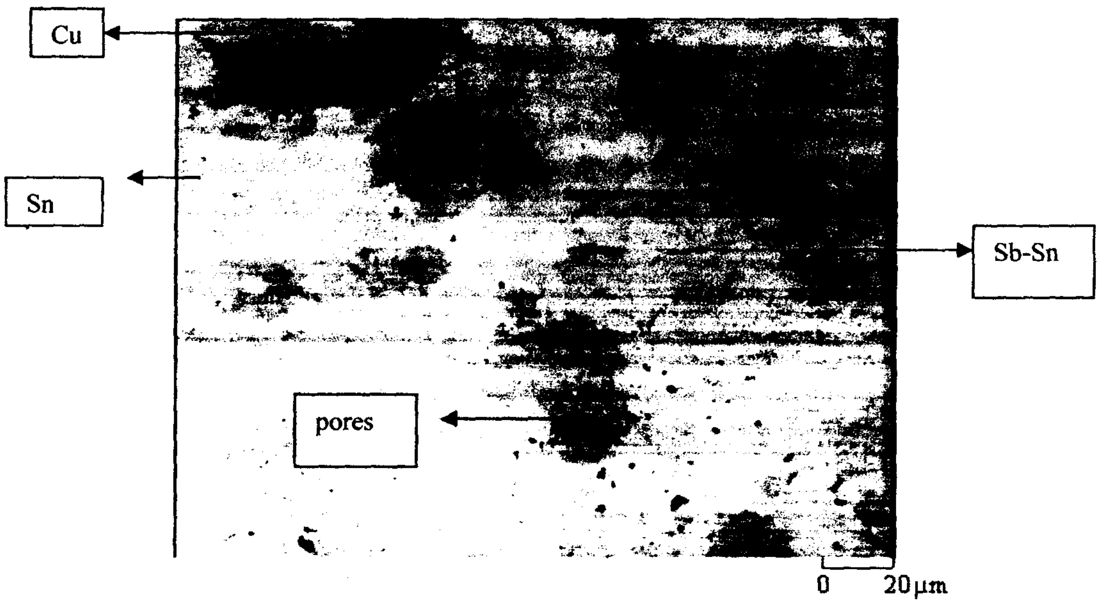


Figure 5.6.4 Microstructure of Experiment 5 (220°C, 156 MPa, microwave, 30 min)

Figure 5.6.5 shows the specimen conventionally sintered at 220°C for 60 minutes and compacted at 156 MPa. The specimen under this condition has swelled with a diameter change of 0.28%. It has the poorest dimensional accuracy. It has 7.074 g/cm^3 density and porosity of 1.76%. However, its hardness is highest at 32.86. Largest number of hard inter-metallic compound phase crystals are formed and more uniformly distributed in the structure, when the compacts are sintered conventionally at higher temperature for a longer period, even when the compaction pressure is small, as is evident from Fig 5.6.5.

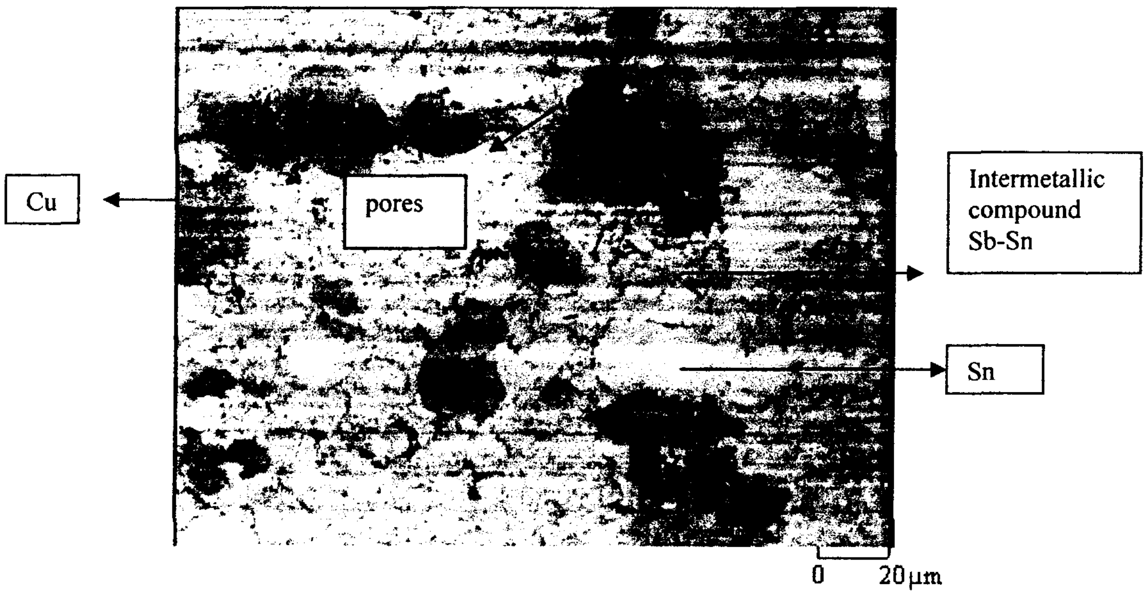


Figure 5.6.5 Microstructure of Experiment 6 (220°C, 156 MPa, Ar gas, 60 min)

Figure 5.6.6 shows the specimen sintered in a microwave furnace at 220°C for 60 minutes and compacted at 156 MPa. The specimen under this condition has swelled with a diameter change of 0.22%. It has 7.177 g/cm^3 density and porosity of 0.4%. Its hardness is 28.21. When microwave sintering is performed on specimens compacted at high pressure, with high temperature, even prolonged heating does not precipitate out the hard inter-metallic phase to the extent as in done in conventional sintering as shown in Figure 5.6.6.

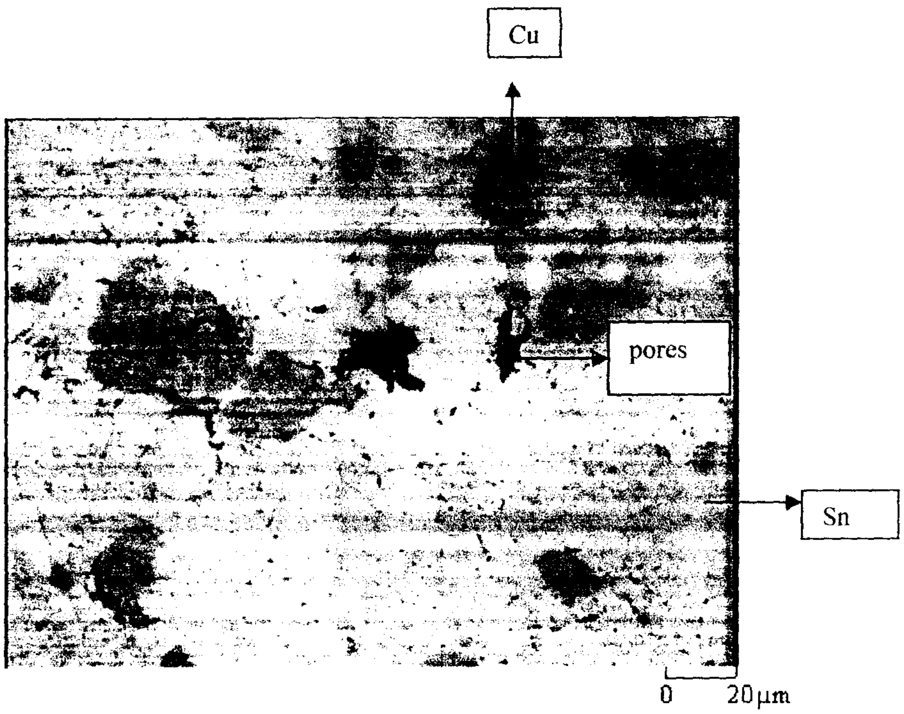


Figure 5.6.6 Microstructure of Experiment 7 (220°C, 312 MPa, microwave, 60 min)

Table 5.3.4.1 continued

Check the significant interactions		C_0	C_1	
	A_0	16.992+17.118 = 34.11	16.139+16.883 = 33.02	A_0C_0 is optimal
	A_1	16.910+16.988 = 33.90	17.017+16.951 = 33.97	
		B_0	B_1	
	A_0	16.992+16.139 = 33.131	17.118+16.883 = 34.001	A_0B_1 is optimal
	A_1	16.910+17.017 = 33.927	16.988+16.951 = 33.939	
		D_0	D_1	
	A_0	16.992+16.883 = 33.875	17.118+16.139 = 33.257	A_1D_1 is optimal
	A_1	16.910+16.951 = 33.861	16.988+17.017 = 34.005	
Optimum combination of significant factor levels	$C_0B_1A_1$			
Remarks	The significant factors are chosen from the left hand side in the above Pareto diagram which cumulatively contribute about 95.7%			
Overall optimum conditions for all factors	A_1 = sintering temperature, 140°C B_1 = compaction pressure, 312MPa C_0 = microwave sintering D_0 = sintering time, 30min			
Estimate of error variance	Ve= (pooled variation of non-significat sources)/ (pooled degrees of freedom of non-significant sources) = 0.2247/8 = 0.0281			

It is observed from Pareto ANOVA (Table 5.3.4.1) that factor B (compaction pressure) and factor C (type of sintering) are the most significant factors affecting the density; factor C contributing 19.8% and factor B contributing 14.9% respectively to the density. This can be seen from Figure 5.3.4.2. Factor A (Sintering temperature) has complex interaction effects with compaction pressure, type of sintering and sintering time. The largest contribution is of interaction between A and C i.e. the sintering temperature and type of sintering (25.6%); where as the interactions of sintering temperature with the compaction pressure and the time of sintering contribute only 14 and 11 % respectively. It is obvious that the higher the compaction pressure the higher will be the green density and higher will be the sintered density (German,1994). In general microwave sintering is observed to give higher density and has quite a high significant effect on density (Figure 5.3.1.1). From the Pareto diagram (Table 5.3.4.1), it is also seen that A (sintering temperature) and D (sintering time) have less significant effect. Therefore, it does not matter if A_0 (140°C) and D_0 (30 minutes) or A_1 (220°C) and D_1 (60 minutes) is chosen. From the iso-level analysis, the optimum combination of input parameters for highest density is $A_1B_1C_0D_0$ (220°C sintering temperature, 312MPa compaction pressure, microwave sintering and 30 minutes sintering time). Therefore, this combination may be considered as the optimum combination.

Thus it can be concluded that microwave sintering gives higher density as compared to conventional sintering. For higher density higher compaction pressure shall be used with less sintering time when sintered with microwave heating.

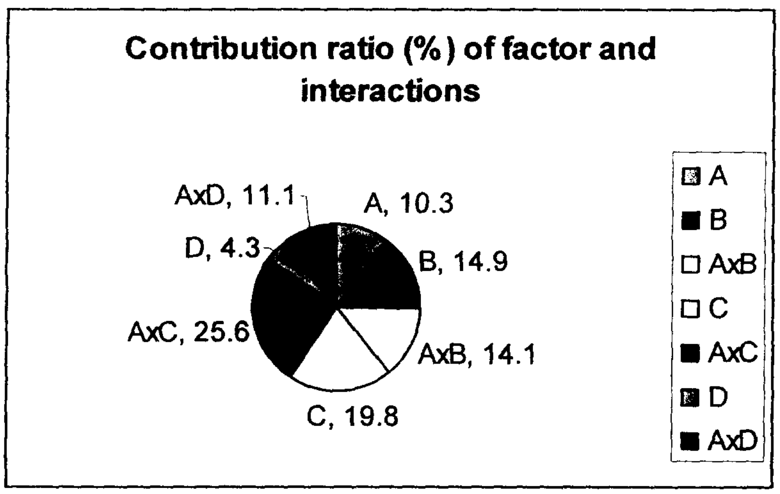


Figure 5.3.4.2 Contribution ratio in percentage of factor and interactions.

5.4 Open pores porosity

There are two types of porosities in sintered specimens. The porosity due to the pores interconnected to each other and extending up to surface is called open pores porosity and the porosity due to pores isolated within the body and that do not appear on the surface is called the closed pores porosity. The closed pores porosity shall be smallest to have the highest density, but for certain applications such as self-lubricating bearings the open pores porosity shall be larger, for larger amount of impregnated oil to be retained inside the pores. Because the tin base alloys are mainly used in fabricating self lubricating bearings, this will be the target function to find the optimum conditions using Pareto ANOVA.

Table 5.4 shows the open pores porosity of each experimental run and the SN ratios. The larger-the-better characteristic is used to calculate the SN ratio since a high porosity is desired. The way to compute SN ratio is:

$$SN_i = -10\log\left(1/n\sum_{j=1}^{n} 1/y_j^2\right) \qquad \text{----------Equation (2)}$$

where n = number of replications

$\sum y_{ij}$= porosity values in each row (i^{th} experimental conditions of control factors)

Sample calculation of SN ratio for the first experimental run is given below:

By referring to Table 5.4, the values are:

$n = 3$; $y_1 = 2.00$; $y_2 = 1.82$ and $y_3 = 1.70$

Therefore, $SN_1 = -10\log [(1/3)(1/(2.00)^2 + 1/(1.82)^2 + 1/(1.70)^2 = 5.239$

Array type	Inner array (L_8)							Observations					
Experiment number	Control factor assignment and *column number*							Raw data: Porosity (%)			Average	Standard Deviation	SN(based on larger-the-better characteristic)
	A	B	AxB	C	AxC	D	AxD	y_1	y_2	y_3			
	1	2	3	4	5	6	7						
1	0	0	0	0	0	0	0	2.00	1.82	1.70	1.84	0.15	5.239
2	0	0	0	1	1	1	1	1.13	1.49	2.70	1.77	0.82	3.402
3	0	1	1	0	0	1	1	0.00	0.00	0.07	0.02	0.04	-18.327
4	0	1	1	1	1	0	0	0.11	0.00	0.00	0.04	0.06	-14.401
5	1	0	1	0	1	0	1	1.77	2.17	1.42	1.79	0.38	4.653
6	1	0	1	1	0	1	0	2.17	1.63	1.48	1.76	0.36	4.579
7	1	1	0	0	1	1	0	0.74	0.21	0.26	0.40	0.29	-11.172
8	1	1	0	1	0	0	1	0.12	0.14	0.27	0.18	0.08	-16.506
	Current condition												

Table 5.4 Porosity of each experimental run and its SN ratio.

5.4.1 The effect of input parameters on open pores porosity

Figure 5.4.1 to Figure 5.4.1.3 shows the effect of temperature, compaction pressure, type of sintering and sintering time on porosity, respectively. From the graphs, it can be concluded that porosity increases as the sintering temperature increases (Figure 5.4.1) while porosity decreases as the compaction pressure increases (Figure 5.4.1.1). Microwave sintering yields higher porosity than conventional sintering (Figure 5.4.1.2). Porosity increases as the sintering time increases (Figure 5.4.1.3).

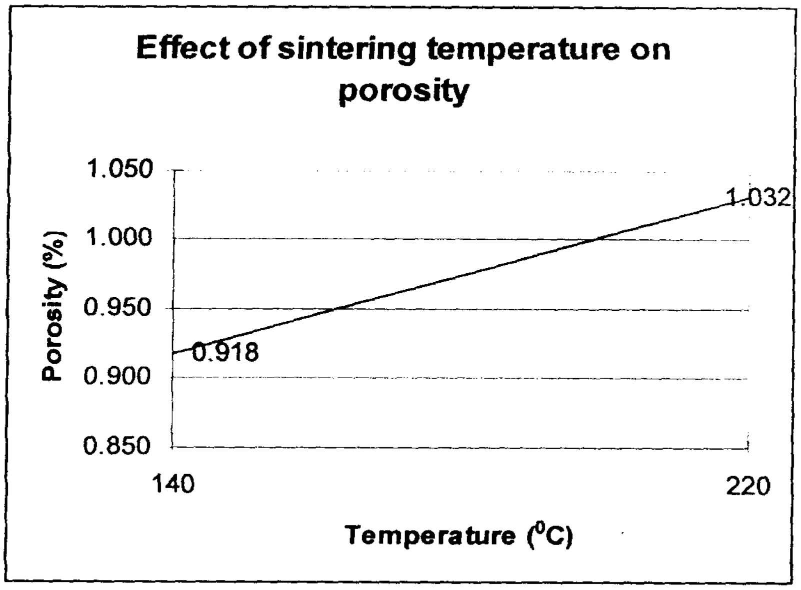


Figure 5.4.1 Effect of sintering temperature on porosity

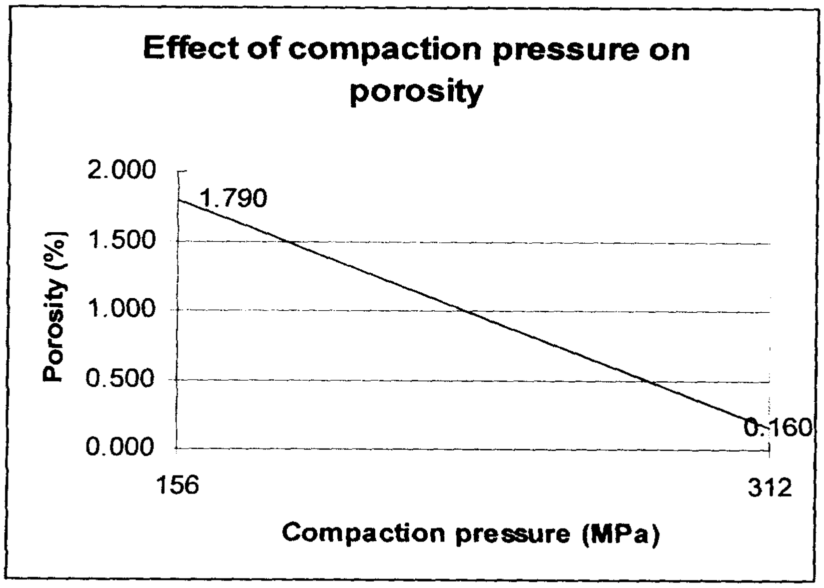


Figure 5.4.1.1 Effect of compaction pressure on porosity

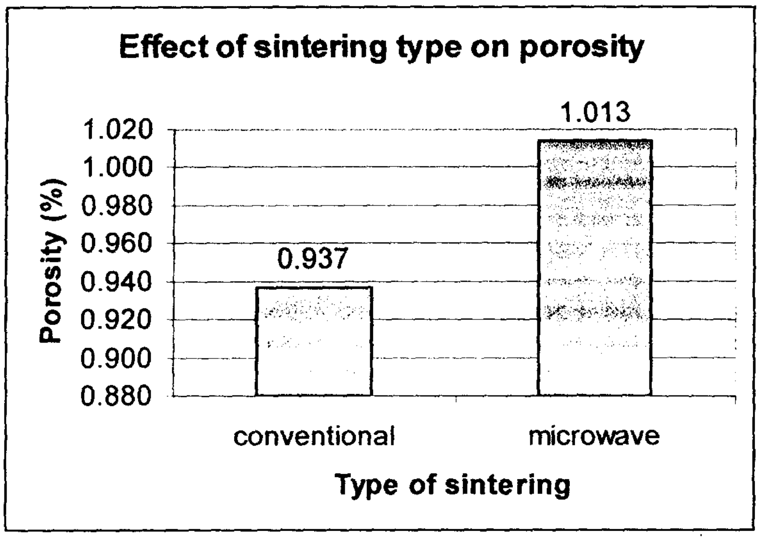


Figure 5.4.1.2 Effect sintering type on porosity

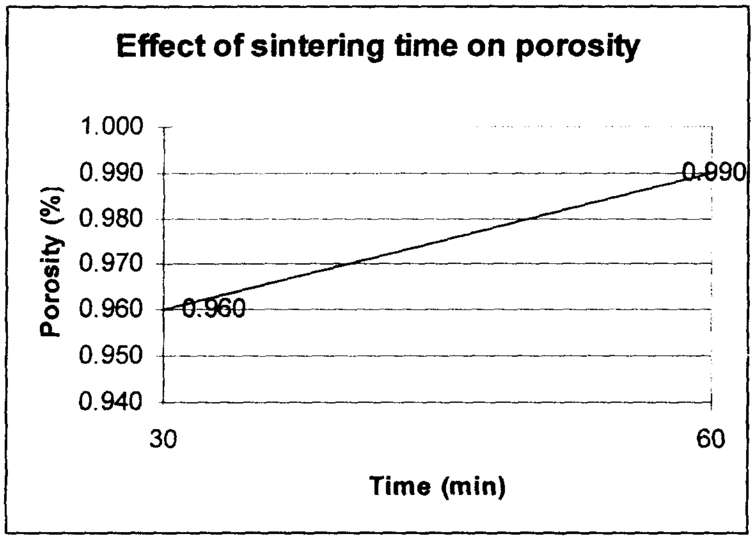


Figure 5.4.1.3 Effect of sintering time on porosity

5.4.2 Pareto Anova

The optimum level for each factor will be at which the average of the "iso-level" value of S/N ratio is maximum. The average iso-level value is determined from the values of S/N ratios at low and high levels of the factors. For example, the average S/N ratio for porosity at low level of factor A is determined from experimental runs 1,2,3 and 4 and for high level from experimental runs 5,6,7 and 8. Similarly, for other factors these iso-levels values are determined and recorded in Table 5.4.2. Figure 5.4.2 shows the iso-level SN values for porosity at different level of parameters.

Factors	**Porosity at Low Level (0)**	**Porosity at High Level (1)**	**Difference**
A Sintering temperature	-6.022	-4.612	1.410
B Compaction pressure	4.468	-15.102	19.570
Interaction AxB	-4.759	-5.874	1.115
C Type of sintering	-4.902	-5.732	0.830
Interaction AxC	-6.254	-4.380	1.874
D Sintering time	-5.254	-5.380	0.126
Interaction AxD	-3.939	-6.695	2.756

Table 5.4.2 Average iso-level SN ratio values for open pores porosity

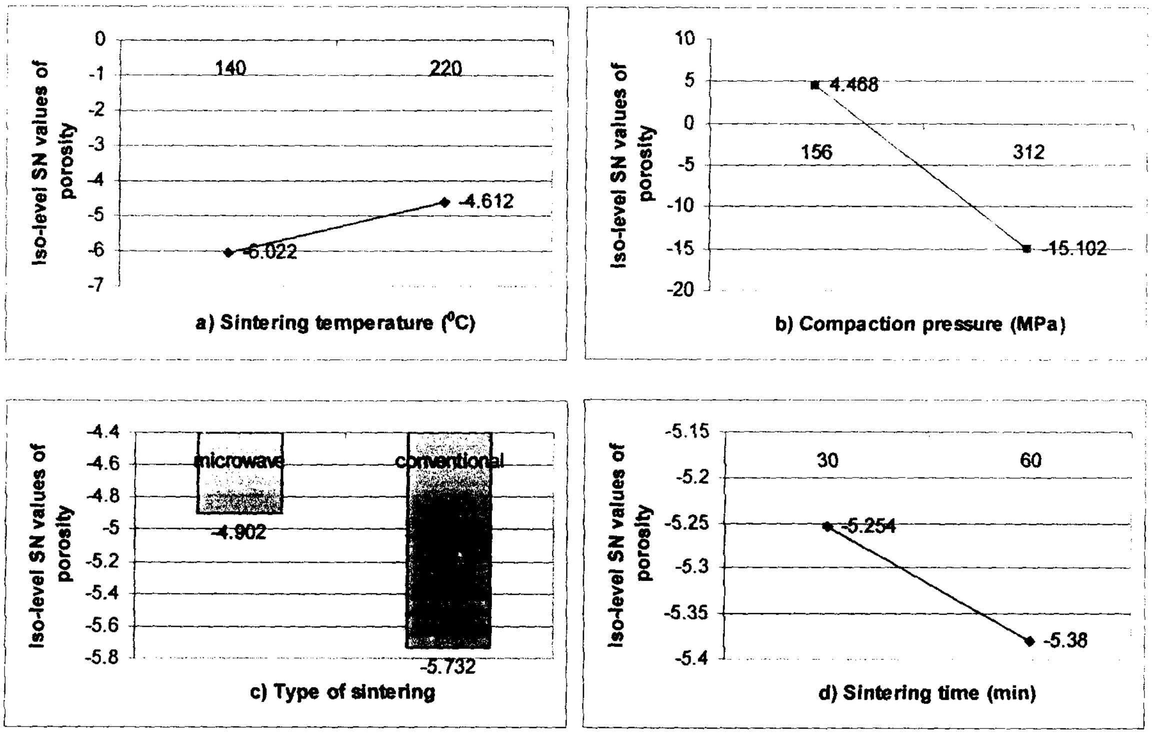


Figure 5.4.2 Iso-level SN values of open pores porosity at different levels of parameter a)sintering temperature; b)compaction pressure; c)type of sintering; d)sintering time

From Table 5.4.2, one can see that the optimum combination of input parameters for highest porosity is $A_1B_0C_0D_0$ (220°C sintering temperature, 156MPa compaction pressure, microwave sintered and 30 minutes sintering time). The Pareto Analysis of Variance (ANOVA) applied to this data gives the same optimum level of controllable factors as the iso-level technique and shown in Table 5.4.2.1.

Table 5.4.2.1 Pareto Analysis of Variance (ANOVA) for porosity

Factor and interactions		A	B	AxB	C	AxC	D	AxD	Total
Sum at factor level	0	-24.087	17.873	-19.037	-19.607	-25.015	-21.015	-15.755	sum of 0,1 level= -42.533
	1	-18.446	-60.406	-23.496	-22.926	-17.518	-21.518	-26.778	
Difference at 2 levels		-5.641	-78.279	-4.459	-3.319	-7.497	0.503	-11.023	
Square of difference		31.821	6127.602	19.883	11.016	56.205	0.253	121.507	6368.286
Contribution ratio (%)		0.50	96.22	0.31	0.17	0.88	0.00	1.91	100.00

Figure 5.4.2.1 Pareto diagram for porosity

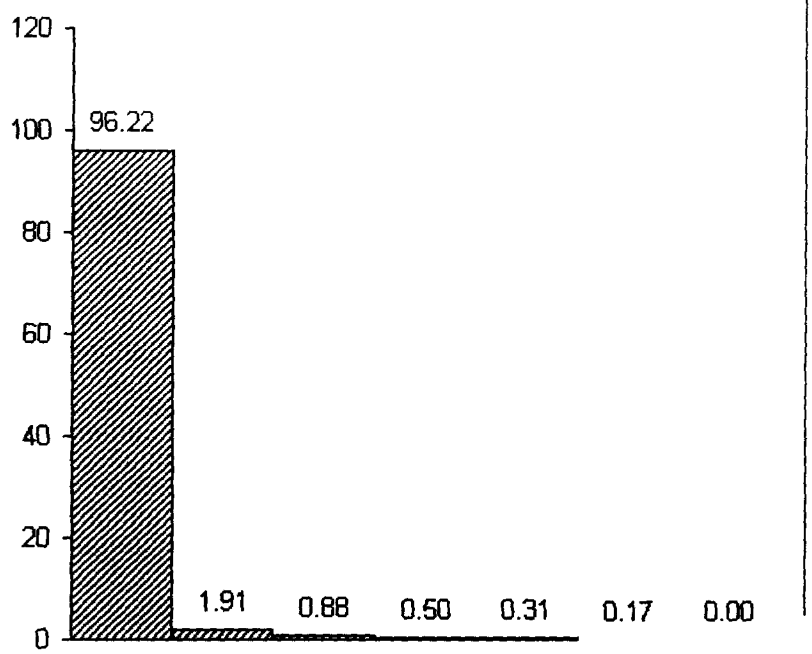


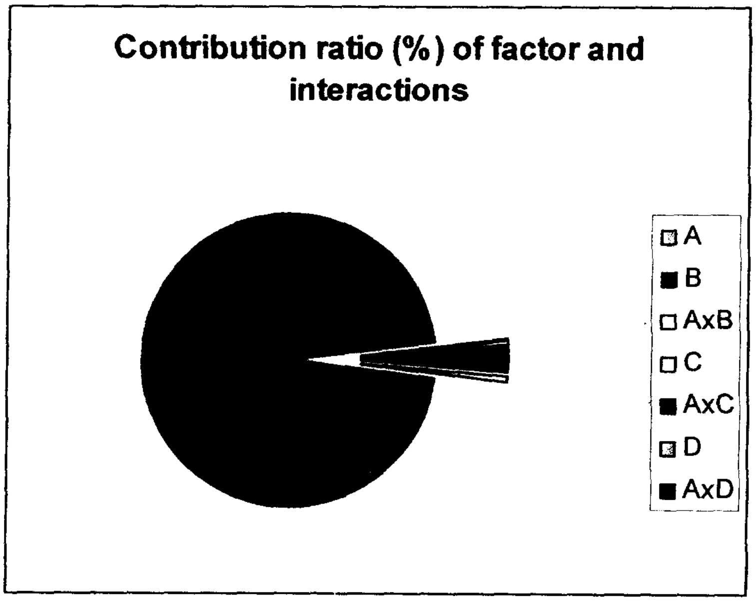
Table 5.4.2.1 continued

Factor and interactions	B	AxD	AxC	A	AxB	C	D
Cumulative contribution ratio (%)	96.22	98.13	99.01	99.51	99.82	99.99	100.00
Optimum combination of significant factor levels	B_0						
Remarks	The significant factors are chosen from the left hand side in the above Pareto diagram which cumulatively contribute about 97.17%						
Overall optimum conditions for all factors	A_1 = sintering temperature, 220°C B_0 = compaction pressure, 156MPa C_0 = microwave sintering D_0 = sintering time, 30min						
Estimate of error variance	Ve= (pooled variation of non-significat sources)/ (pooled degrees of freedom of non-significant sources) = (31.8209/8 + 11.0158/8+ 0.253/8)/3 = 1.7954						

From Pareto ANOVA given in Table 5.4.2 it is seen that the most significant factor, affecting open pore porosity, is factor B (the compaction pressure). It contributes 96 % to this characteristic as shown in Figure 5.4.2.2. The lower the level of compaction pressure the higher the open pores porosity, because at lower pressures the green compact density is low, and the green compact contains lots of pores, which remain even after sintering .

However, from Table 5.4.1, one can see that the optimum combination of input parameters for highest porosity is $A_1B_0C_0D_0$, within the tested range, i.e. microwave sintering at temperature 220°C, compaction pressure 156 MPa and sintering time 30 min.

Thus, it can be concluded that microwave sintering gives slightly higher porosity than the conventional sintering, if low compaction pressure, high sintering temperature and low sintering time is used.



A= 0.5%, B = 96.2%, Ax B = 0.3%, C = 0.2%, Ax C = 0.9%, D = 0% and Ax D = 1.9%

Figure 5.4.2.2 Contribution ratio in percentage of factor and interactions

5.5 Vickers Hardness Test

Table 5.5 shows the Vickers hardness number (VHN) of each experimental run and the SN ratio. The larger-the-better characteristic is used to calculate the SN ratio. The way to compute SN ratio is:

$$SN_i = -10\log\left(1/n\sum_{j=1}^{n} 1/y_{ij}^2\right) \qquad \text{----------Equation (2)}$$

where n = number of experiment

$\sum y_{ij}$= hardness value in each row (ith experimental conditions of control factors)

Sample calculation of SN ratio for the first experimental run is given below:

By referring to Table 5.5, the values are:

n = 3; y_1 = 21.367; y_2= 21.633 and y_3 = 21.567

Therefore, $SN_1 = -10\log [(1/3) (1 / (21.367)^2 + 1/ (21.633)^2 + 1/ (21.567)^2 = 26.657$

Array type	Inner array (L_8)							Observations					
	Control factor assignment and *column number*							Raw data: VHN			Average	Standard Deviation	SN(based on larger-the-better characteristic)
	A	B	AxB	C	AxC	D	AxD	y_1	y_2	y_3			
Experiment number	1	2	3	4	5	6	7						
1	0	0	0	0	0	0	0	21.367	21.633	21.567	21.522	0.139	26.657
2	0	0	0	1	1	1	1	25.200	23.733	24.100	24.344	0.763	27.720
3	0	1	1	0	0	1	1	21.967	22.100	22.600	22.222	0.334	26.934
4	0	1	1	1	1	0	0	24.267	22.733	23.733	23.578	0.779	27.440
5	1	0	1	0	1	0	1	24.900	22.733	24.500	24.044	1.153	27.600
6	1	0	1	1	0	1	0	32.800	32.100	33.667	32.856	0.785	30.327
7	1	1	0	0	1	1	0	26.267	26.733	31.633	28.211	2.973	28.919
8	1	1	0	1	0	0	1	26.700	26.833	26.633	26.722	0.102	28.537
	Current condition												

Table 5.5 Hardness of each experimental run and its SN ratio.

5.5.1 The effect of input parameters on hardness

Figure 5.5.1 to Figure 5.5.1.3 shows the effect of temperature, compaction pressure, type of sintering and sintering time on hardness, respectively. From the graphs, it can be concluded that hardness increases as the sintering temperature increases (Figure 5.5.1) while hardness decreases as the compaction pressure increases (Figure 5.5.1.1). Conventional sintering yields higher hardness than microwave sintering (Figure 5.5.1.2). Hardness increases as the sintering time increases (Figure 5.5.1.3).

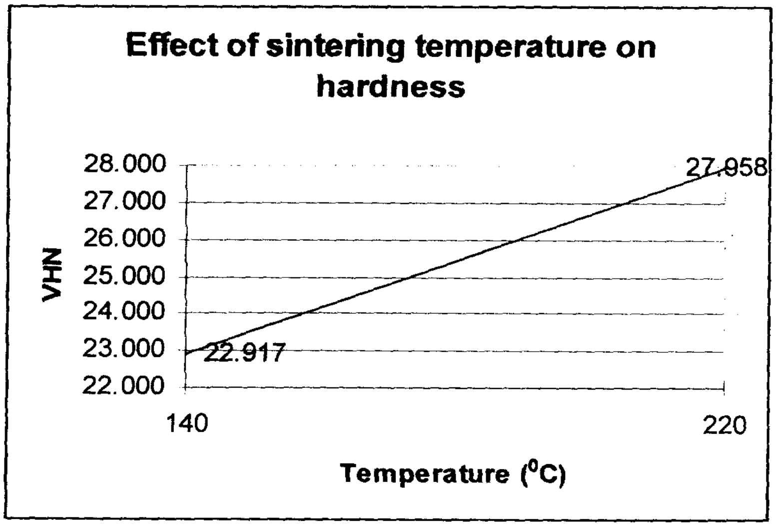


Figure 5.5.1 Effect of sintering temperature on hardness

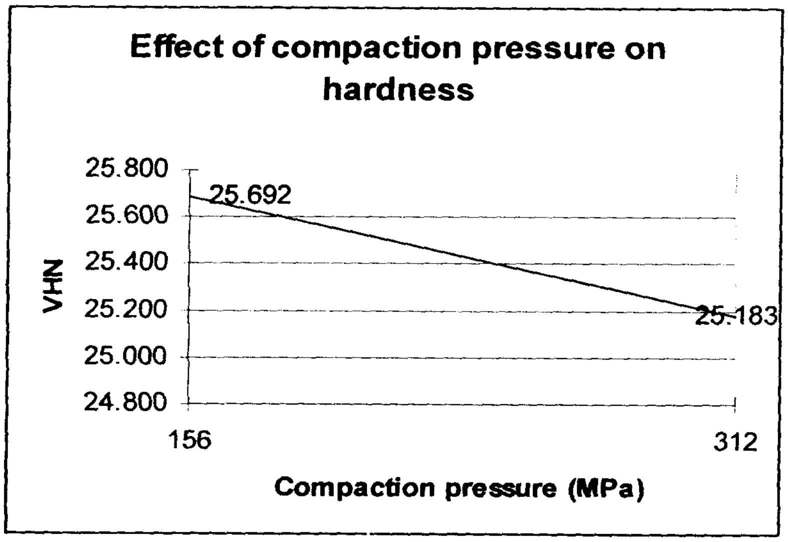


Figure 5.5.1.1 Effect of compaction pressure on hardness