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Understanding the Strength of Hot-Pressed Nanostructured Powder Compacts

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Attrition-milled nanostructured powders were hot pressed, and macroscopic properties of density, hardness, grain size, and strength were measured. No correlation was found between processing conditions (temperature and time) used in this study and compact properties, nor was a correlation found between the tensile (or failure) stress and density, hardness, or grain size. Variations of compact properties of unmilled powder were similar to that of milled powders. Tensile data were not well fitted to a Gaussian distribution but were well fitted to a two-parameter Weibull distribution. Thus, although the milled powder compacts had an average tensile strength greater than the unmilled powder compacts, all sample compositions fit a distribution with zero as a possible minimal stress level. Weibull analysis suggests that the tensile and compression strength is controlled by the presence of fine cracks, which may limit future engineering applications. Efforts to eliminate these cracks during hot pressing were unsuccessful.

Keywords compact, nanostructure, properties, statistics, strength, Weibull distributions

1. Introduction

Before a material can be put into engineering service, its material performance must be determined. Engineering applications of a material require that properties of a product be reliably reproduced and the expected variation known. Recently, interest in nanostructured materials has expanded from developing processing techniques and characterization of the nanostructure of a powder (Ref 1) to: (a) developing processing techniques for compaction of nanostructured powders, (b) production of macroscopic components, and (c) evaluation of the macroscopic properties of the compact (Ref 2-9). In this study, the strength of hot-pressed compacts made from attrition-milled nanostructured powders was studied. In addition to evaluating the strength and strength variability of the compacts, properties that can be evaluated nondestructively were measured and correlated to the strength of the compacts.

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Strength of powder compacts depends upon: (a) the type of particle bonding that formed during compaction, (b) the amount of interparticle bonding achieved, and (c) the effect of impurities present in the bond interface, etc. (Ref 10). However, these characteristics are difficult to measure because they are microscopic and can vary throughout the compact and generally require destructive testing for evaluation. In this study, nondestructive compact properties were evaluated and compared to compact strengths. To help understand the parameters affecting strength and variability in strength values, individual compact parameters (density, hardness, and grain size) were measured, and the processing temperatures and times were varied. Both the variations in processing conditions and variations in compact properties were correlated to strength. Failure characterization herein includes not only the determination of the central measures of the scatter in strength behavior of the material (e.g., mean and variance), but also the estimation of the cumulative distribution function.

2. Experimentation

Attrition-milled iron alloyed powders were hot pressed and tensile tested. Characterization of the starting powders is shown in Table 1. The powders were processed in attrition mills

Table 1 Starting attrition-milled characteristics

Composition	Surface area, m^2/g	Particle size, μm	Grain size, nm	Oxygen, wt%
Fe	0.36	29	>1000	0.47
Fe-[Ar]	0.57 ± 0.16	8.5 ± 1.5	7.2 ± 0.7	2.76 ± 1.1
Fe-2Al	0.47 ± 0.21	10.2 ± 2.9	8.1 ± 1.4	2.92 ± 1.3
Fe-2C	0.47 ± 0.17	8.4 ± 1.5	4.9 ± 0.6	3.83 ± 1.6
Fe-2Al-2C	0.52	10.2	4.8	2.87
Fe-5Al	0.35 ± 0.11	11.6 ± 1.7	9.0 ± 0.9	2.01 ± 0.5
Average	0.48 ± 0.08	9.8 ± 1.3	6.8 ± 1.9	2.87 ± 0.7

Note: Properties of individual runs and compositions can be found in Ref 5-8.

for 150 h using argon as the cover gas. Five different compositions were produced: (a) iron, Fe-[Ar]; (b) iron alloyed with 2 wt% Al, Fe-2Al; (c) iron alloyed with 2 wt% C, Fe-2C; (d) iron alloyed with 2 wt% Al and 2 wt% C, Fe-2Al-2C; and (e) iron processed with 5 wt% Al, Fe-5Al. In addition to the milled powder, as-received iron powder, Fe-(as-rec), was used to provide a baseline against which the properties of milled powders could be compared. Details of the milling process and complete characterization of the milled powders can be found in Ref 5, 6, 11, and 12.

Tensile samples were made by hot pressing approximately 10 g of milled powder in a graphite die in a vacuum induction furnace. All powders were compacted under 50 MPa, the maximum pressure possible for the graphite dies used in this study. Extended description of hot-pressing procedures can be found in Ref 3 and 6. Processing temperatures varied from 700 to 900 °C and processing times from 1 to 100 min. For most time-temperature combinations, duplicate compaction samples were made. (See Table 2 for an example of run conditions.) Compacts were disks 20 mm in diameter by 7 to 10 mm thick. The hot-pressed samples were metallurgically polished on both sides and characterized by x-ray diffraction to determine the phases present and the grain size. Hot-pressed sample characterization also included immersion density and hardness (HRC). For the Fe-5Al powder, 24 hot-pressed samples were made under identical time-temperature processing conditions (Table 3). Complete description of the Fe-5Al powder, compaction, and test results can be found in Ref 13.

Tensile sample configuration was made from the compacts by cutting a 7 mm slit in from both sides along the diameter, resulting in an hourglass sample shape, with a tensile stress test region approximately 5 mm wide, 5 mm long, and 5 mm thick. All samples were tested at room temperature. All specimens displayed brittle fracture in that the tensile stress-strain curves indicated limited ductility. Consequently, the tensile failure stress or maximum stress is the focus of this study. In addition, compaction samples 5 by 5 by 10 mm were machined from the grip section of the failed Fe-5Al tensile samples.

3. Data and Analysis

Analyses were conducted in several steps:

1. The starting powders were characterized for particle size, surface area, grain size, chemical composition, and phase.
2. Twenty-four samples compacted from a single powder composition and the same attritor run were hot pressed under identical conditions and analyzed with respect to the characteristics of individual compact density, hardness, grain size, and tensile (and compression) strength.
3. Hot-pressed samples prepared under different temperatures and times were characterized with respect to compact physical properties and different starting powder lots.
4. Strength characteristics for different milled compositions were compared.

To simplify the statistical analysis, the same sample geometry was used for all tensile and compression strength tests.

Statistical analyses included (a) characterization of an individual parameter by determining the average, standard deviation, and frequency distributions (both Gaussian and Weibull) of both starting powders and compact properties, (b) correlations between parameters (e.g., Pearson correlations between compact properties), and (c) correlation within parameters (e.g., strength as a function of compact density).

Table 2 Hot-press processing temperature and time conditions and resulting compact density, grain size, hardness, and tensile data for Fe-2Al-[Ar] samples

Temperature, °C	Time, min	Density, g/cm^3	Grain size, nm	Hardness, HRC	Tensile strength, MPa
700	25	7.26	267	42	359
800	10	7.06	84	46	425
		6.79	91	43	212
		7.03	29	46	542
		7.16	50	48	504
		7.05	136	49	249
800	25	7.25	33	50	743
		6.92	27	37	216
		7.17	42	50	497
		7.13	30	52	424
		7.05	160	45	580
800	50	7.25	32	50	724
		7.13	155	49	782
800	100	7.30	30	50	809
		7.15	95	49	899
		7.14	98	48	842
850	100	7.33	34	49	980
900	3	7.33	50	46	956
		7.15	56	44	533
900	10	7.19	262	40	203
900	25	7.19	206	44	619
		7.31	32	32	333

The starting powders were all processed in attrition mills under similar processing conditions. For most of the compositions, several different powder milling lots were used to prepare the hot-pressed compacts. After 150 h of milling, the measured variation in powder properties between milling lots for the same composition were less than the measured variation within the same powder lots. For a given milled composition, the milled surface area, particle size, and oxygen concentration for all powder compositions were nearly identical. Attrition milling produced particles with two grain sizes, approximately 8 nm for powder compositions without carbon and 5 nm for powder compositions with carbon. For further characterization of the starting powders, see Ref 13. Thus, for a given composition, the starting powder for the hot-press compacts was considered to be similar (Table 1).

Before discussion of strength characterization of the hot-press compacts, it should be noted that in general the hot-press conditions used in this study produced compact densities of >98% of theoretical density. For the hot-pressed Fe-(as-rec) and Fe-[Ar] compacts, the average density was >99%. The hot-pressed Fe-5Al compacts had an average of approximately 95% of theoretical density. The temperature and processing times in this study resulted in higher densities than previously reported (Ref 3, 4) for strength characterization of nanostructured powder samples, but the higher processing temperatures and times used in this study also resulted in larger grain sizes than many compacted nanostructure studies.

The oxygen and the carbon concentrations of the milled powder reported in Table 1 came out of solution and reacted with the iron powder to form grain precipitates less than 1 μm diameter that were uniformly distributed throughout the compacts (Ref 11). The precipitate distribution and the precipitate grain size did not vary with the oxide (and/or the carbide) concentration or with the different compact processing conditions used in this study. Neither had an effect on the compact strength, nor did the precipitate distribution and the precipitate grain size effect the compact strength.

To study hot-press process reproducibility, 24 samples were prepared under similar conditions: 800 °C, 15 min hold time, and 50 MPa using the same starting powder, Fe-5Al. Compact properties showed little variation between samples (Table 3). The percent standard deviation for density was 1.6; for hardness, 4.2; and for grain size, 4.8. Two additional samples were made later in the study, again under identical processing conditions, and compared to the original 24 samples. The reproducibility of compacts throughout the entire period of this study shows that neither the hot press equipment nor the operating procedures had changed over time.

Tensile and compaction tests of the compacts showed that these samples had very little ductility and that the yield, tensile (or maximum), and failure stresses were approximately the same. Because the three stresses were approximately equal, only a single stress value was reported and referred to as tensile stress.

For powder metallurgy (P/M) compacts, there is a strong correlation between tensile strength and density (Ref 10). For cast metals, there is a strong correlation between tensile strength and grain size (Hall-Petch relationship) and between tensile stress and hardness (Ref 14). For the Fe-5Al nanostructured compacts formed under identical processing conditions, no correlation was found between tensile and compression strength values and density, grain size, or hardness. Correlation plots show a random distribution and no statistically significant Pearson correlation was found (Fig. 1). Although the compression failure stress was approximately 200 MPa greater than the tensile stress, both stresses had a similar Weibull distribution shape factor, η (see Appendix for derivation and explanation of η), suggesting they failed by similar mechanisms. Also, no correlation was found between tensile and compression strengths for the same sample.

In addition to the hot-press study in which only one processing condition was used, hot-press studies were conducted varying the processing temperatures and times. To determine the variability between and within a temperature-time processing condition, multiple samples were produced for most processing conditions (Table 2, 4).

In general, for a given powder composition, density, hardness, and tensile values for an individual temperature-time processing condition were within one standard deviation of the average value when all processing conditions were considered (Table 5). Also, there was little difference in the density, hardness, and tensile strength standard deviation for the 24 samples compacted and the standard deviation of the duplicate multiple temperature and time data for each composition. For example, the data for density variation range from ±0.09 for Fe-5Al, ±0.05 for Fe-(as rec), ±0.08 for Fe-[Ar], and ±0.10 for Fe-2Al

Table 3 Hot-press data for Fe-5Al samples all processed under similar temperature (800 °C), time (15 min), and pressure (50 MPa)

Density, g/cm^3	Hardness, HRC	Grain size, nm	Tensile, MPa	Compression, MPa
5.741	88.00	156	290	BDM(a)
5.522	97.50	148	194	BDM
5.567	91.75	159	294	BDM
5.387	94.25	151	319	432
5.360	87.00	154	168	BDM
5.437	94.00	154	262	BDM
5.541	98.25	154	231	619
5.521	90.25	158	339	BDM
5.566	97.75	156	BDM	BDM
5.505	89.00	169	321	435
5.527	90.75	158	BDM	BDM
5.585	96.25	168	329	446
5.671	97.00	174	BDM	718
5.640	90.50	171	296	599
5.717	94.75	172	382	551
5.548	98.00	164	BDM	424
5.571	94.75	166	174	BDM
5.569	98.75	159	312	BDM
5.466	89.50	170	BDM	BDM
5.570	89.75	177	255	417
5.538	90.25	168	351	216 & 519
5.571	97.75	168	BDM	274
5.518	97.50	161	205	BDM
5.678	99.25	164	438	521
5.510(b)	94.10	157	302	…
5.530	87.25	168	211	…

The first 24 samples were from a duplication study and were all processed in succession. (a) BDM, broke during machining. (b) The data sets in the bottom two rows are from hot-press runs at the beginning and end of this study.

(Table 5). Because the data can be considered as a single statistical population regardless of processing conditions, estimations for the compact density and hardness can be obtained from fewer samples than otherwise anticipated. Data from different processing conditions can be combined with caution to improve statistical analysis.

Table 4 Gaussian and Weibull analysis for tensile and compression stress data

	Fe-as received	Fe-[Ar]	Fe-2Al	Fe-C	Fe-Al-C	Fe-5Al-(T)	Fe-5A1-(C)
Tensile/compression, MPa	617, 511, 494, 311, 497, 526, 448, 490, 412, 532, 435, 421, 269, 368, 371	554, 510, 323, 681, 460, 340, 923, 539, 769, 267, 184, 726, 337, 472, 516, 598, 604, 398, 632, 1086, 396, 1053, 691, 126	359, 425, 542, 504, 249, 743, 216, 497, 424, 580, 724, 809, 980, 956, 533, 203, 619, 333, 842, 899, 782	76, 125, 94, 276, 234, 295, 432, 290, 470, 165, 551, 801, 474, 671, 312, 598, 137, 568, 624, 672, 448, 900, 398, 129	46, 562, 150, 63, 206 288, 232, 272, 527, 266, 131, 300, 112	290, 194, 294, 319, 168, 262, 231, 339, 321, 329, 296, 382, 174, 312, 255, 351, 205, 438	432, 619, 435, 446, 718, 599, 551, 424, 417, 313, 519, 274, 521
General statistics							
No. samples	15	24	21	24	13	18	13
Average	446.8	549.4	581.9	405.8	242.7	286.7	482.2
Standard deviation	91.5	247.3	242.1	233.8	158.1	72.9	122.9
Coefficient of variation	20.5	45.0	41.6	57.6	65.1	25.4	25.5
Weibull statistics							
Shape, η	5.82	2.42	2.72	1.84	1.67	4.51	4.49
Scale, x_m	482.5	620.2	656.0	457.2	272.5	314.1	528.2
Average	446.9	549.9	583.5	406.2	243.5	286.7	482.0
Standard deviation	89.1	241.9	231.5	229.2	149.9	72.2	121.7
Coefficient of variation	19.9	44.0	39.7	56.4	61.6	25.2	25.3
Kologorov-Smironov	0.11	0.06	0.08	0.11	0.16	0.09	0.17

Table 5 Comparison of individual averages from duplicate temperature-time runs and the average for all runs irrespective of processing conditions

Temperature, °C	Time minutes	No. of samples	Density, g/cm^3	Hardness, HRC	Tensile strength, MPa
Fe-(as received)					
700	25	3	7.69±0.03	B-85±2	541±67
800	10	3	7.75±0.03	B-85±4	487±55
	25	4	7.77±0.03	B-87±2	460±64
900	3	2	7.78±0.02	B-60±1	345±107
	10	2	7.74±0.01	B-63±2	370±21
Average		15	<7.74±0.05>	<B-79±13>	<437±92>
Fe-[Ar]					
700	25	2	7.47±0.00	48±4	532±31
800	10	3	7.51±0.03	42±3	574±308
	25	4	7.41±0.03	37±8	487±304
900	3	3	7.48±0.03	31±7	533±117
Average		24	<7.46±0.08>	<33±10>	<540±248>
Fe-2Al-[Ar]					
800	10	5	7.07±0.03	48±1	432±159
	25	5	7.13±0.10	50±2	561±137
	100	3	7.20±0.09	49±1	850±46
900	3	2	7.24±0.13	45±1	744±219
	25	2	7.25±0.06	49±1	476±202
Average		21	<7.17±0.10>	<46±5>	<582±242>
Fe-2C-[Ar]					
700	25	2	7.08±0.02	50±3	441±276
800	10	3	7.28±0.03	42±3	308±153
	25	2	7.24±0.04	39±5	676±176
900	3	2	7.25±0.02	41±2	455±202
	10	2	7.29±0.03	33±2	353±304
Average		24	<7.24±0.08>	<41±8>	<406±234>
Fe-5Al-[Ar]					
800	15	24	<5.55±0.09>	<93±4>	<287±73>

When the tensile data are examined, however, it is apparent that the scatter in the data is more appreciable. As seen in Table 4, the sample coefficients of variation range from 20 to 65%. With that degree of scatter in the data, statistically meaningful analysis can be obtained only from a large number of replicated tests. Thus, some care must be given to whether or not the tensile failure stress data from samples prepared under different processing conditions can be merged into a single statistical sample. Figures 2, 3, and 5 show the sample average and standard deviations of the tensile stress, density, and grain size, respectively, from different processing conditions along with the merged population. There was little difference in the statistical spread between an individual processing condition for the Fe-5Al and that from the merged data from all the other processing conditions for the rest of the compositions (except for the possible Fe-Al density distribution, which might be a statistical anomaly). As with the Fe-5Al data, when using all of the merged data, no correlation between the tensile stress and the compaction density, hardness, and grain size is observed (Table 6). The plot of the correlation between the compact physical properties and the tensile failure stress (Fig. 2) indicates the same degree of scatter as was observed for the Fe-5Al data (Fig. 1). Thus, with a careful examination, data from different studies were found to be sufficiently similar and could, thus, be combined for statistical analyses.

Both Gaussian and Weibull statistics were used to characterize the tensile strength data (Table 6). The average tensile values determined using Gaussian statistics for the attrition-milled and compacted Fe-[Ar] and Fe-2Al powder were approximately 100 MPa greater than that of the compacted as-received powder, Fe-(as rec). However, there was also a significant increase in the standard deviation for the compacted milled powders. The tensile data has a considerable amount of skew, which suggests that Gaussian distribution may not be the most appropriate method of analysis. While the Gaussian distribution may be more widely used and reported for strength analysis (and for material property analysis in general), the physics used to describe tensile strength, especially powder compact strength, is better represented by the Weibull distribution. Gaussian distribution assumes a data range over all positive and negative real numbers, which is not possible for tensile properties. Weibull statistics have a distribution that starts at some positive value below which no value is possible.

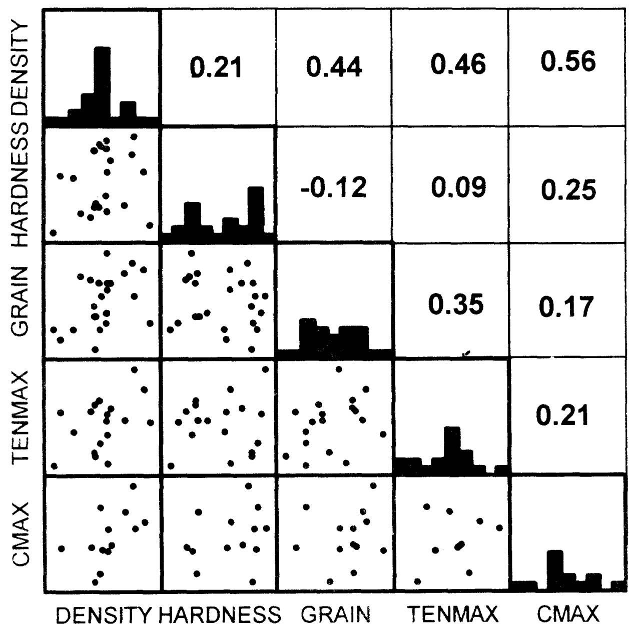


Fig. 1 Correlation plots and Pearson correlations among density, hardness, grain size, maximum tensile stress (tenmax), and maximum compression stress (cmax) for hot-pressed Fe-5Al prepared under similar temperature-time conditions

Table 6 Average, standard deviation, and correlation statistics for strength, density, hardness, and grain size for the hot press compacts

Composition	Fe-as received	Fe-[Ar]	Fe-2Al	Fe-C	Fe-Al-C	Fe-5Al-(T)	Fe-5Al-(C)
Strength, Mpa							
Average	437	540	582	406	243	287	482
% std. dev.	±92	±248	±242	æ234	±158	±73	±123
Density, g/cm^3							
Average	7.74	7.46	7.17	7.24	6.76	5.55	5.55
% std. dev.	±0.5	±1.1	±1.5	±1.7	±1.6	±1.6	±1.6
Hardness, HRC							
Average	79*	33	46	41	47	94	94
% std. dev.	±13	±37	±11	±20	±16	±4.2	±4.2
Grain size, nm							
Average	378	314	85	273	157	162	162
% std. dev.	±97	±93	±87	±98	±68	±4.3	±4.3
Correlations							
tens-dens	–0.41	0.48	0.63	0.27	0.08	0.46	0.56
tens-hard	0.59	0.13	0.18	–0.34	–0.32	0.09	0.25
dens-hard	–0.28	0.12	0.04	–0.45	0.52	0.21	0.21
tens-grain	0.53	0.24	–0.33	0.42	–0.09	0.35	0.17

Temperature (°C)	Time (min.)	No. of Samples	
700	25	3	Fe-(As Rec.)
800	10	3	
	25	4	
900	3	2	
	10	2	
700	25	2	Fe – [Ar]
800	10	3	
	25	4	
900	3	3	
	10	2	
	25	2	
800	10	5	Fe – Al
	25	5	
	100	3	
	3	2	
900	25	2	
700	25	2	Fe – C
800	10	3	
	25	2	
900	3	2	
	10	2	
950	1	2	
800	15	24	Fe – 5Al

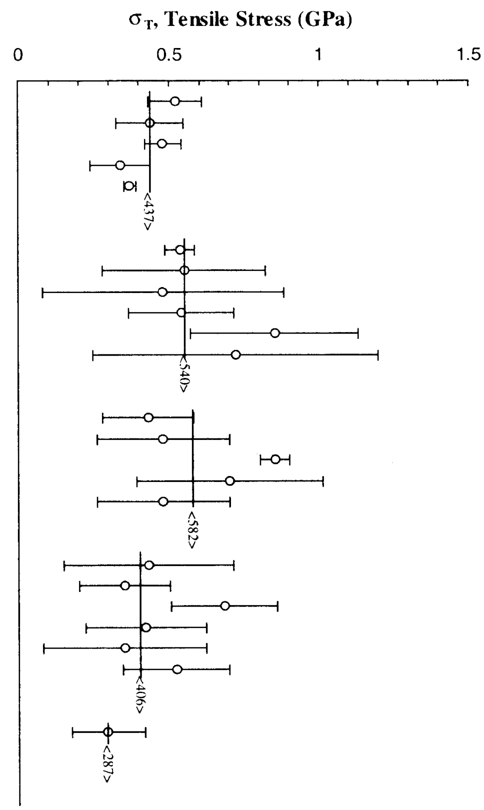


Fig. 2 Plot of the average and standard deviation of the tensile stress for duplicate temperature-time runs, and the average and standard deviation for all the hot-press runs for several hot-pressed compositions

Density (g/cm^3)	No. of Samples	
<7.70	1	Fe-(As Rec.)
7.70-7.725	4	
7.725-7.750	5	
7.750-7.775	4	
>7.775	2	
<7.45	5	Fe – [Ar]
7.45-7.50	10	
7.50-7.55	5	
>7.55	5	
<7.10	5	Fe – Al
7.10-7.20	10	
7.20-7.30	6	
>7.30	3	
<7.20	4	Fe – C
7.20-7.30	12	
7.30-7.40	4	
>7.40	4	
<5.40	2	Fe – 5Al
5.40-5.50	2	
5.50-5.60	15	
5.60-5.70	3	
>5.70	3	

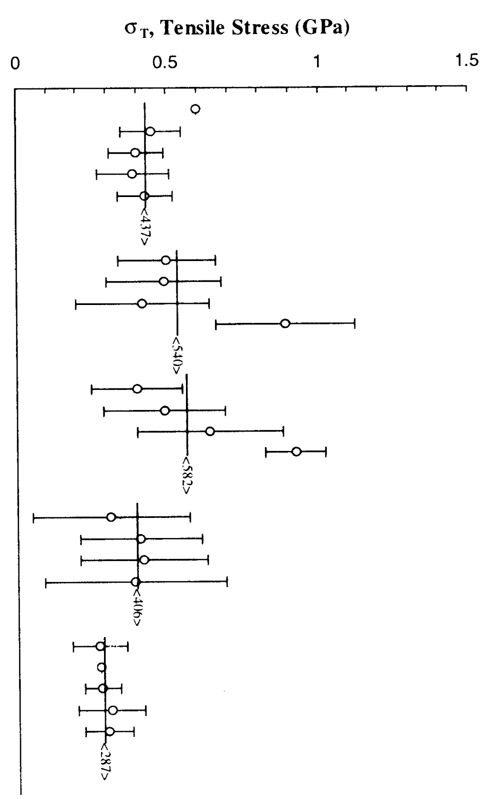


Fig. 3 Plot of the average and standard deviation of tensile stress as a function of density, and the average and standard deviation for all the hot-press runs for several compositions

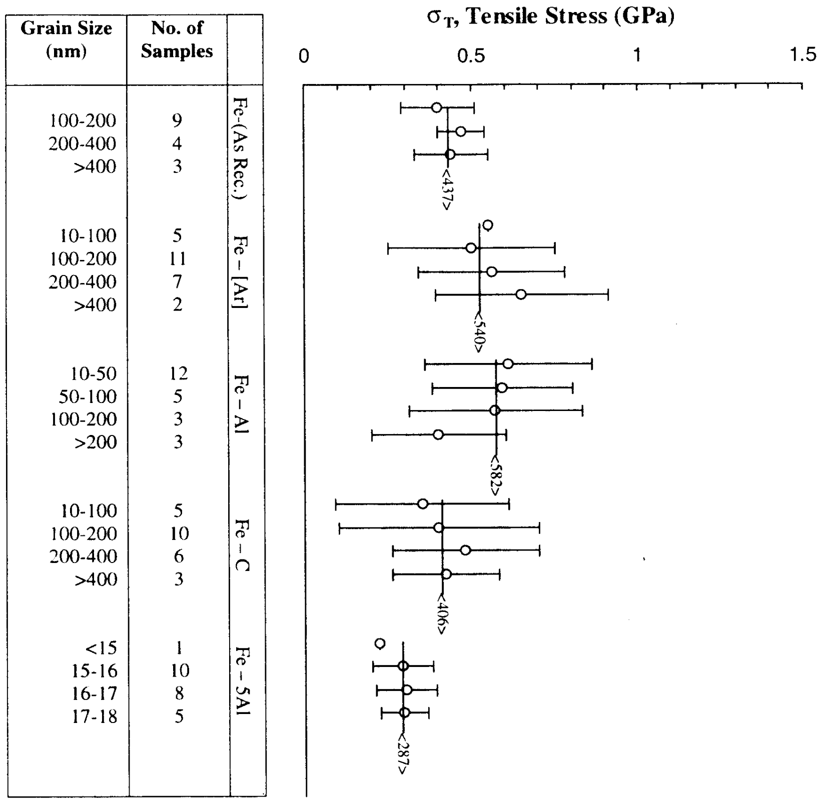


Fig. 4 Plot of the average and standard deviation of tensile stress as a function of grain size and the average and standard deviation for all hot-press runs for several compositions

To illustrate the use of the Weibull cumulative distribution function (cdf), Fig. 5 is a graph of the tensile and compressive failure stress for Fe-5Al, as given in Table 3. The data are plotted on Weibull probability paper, that is, the axes have been normalized so that a Weibull distribution cdf is linear (Ref 14 and Appendix). Thus, if the data are linear, then a Weibull cdf is well suited to represent the data. For these values, a Weibull cdf is an excellent choice for the data. To confirm this observation, the Kologorov-Smironov goodness-of-fit test was applied. The appropriate test statistics for these plots are 0.08 and 0.17, as listed in Table 4. The Weibull cdf would not be rejected unless the value exceeded 0.20. The similarity in slope of the linear fits suggests that the failure mechanisms are identical for the two tests. The shift in the Weibull cdf lines on the graph shows the compressive stresses to be about 1.7 times larger (approximately 200 MPa) than the tensile stresses.

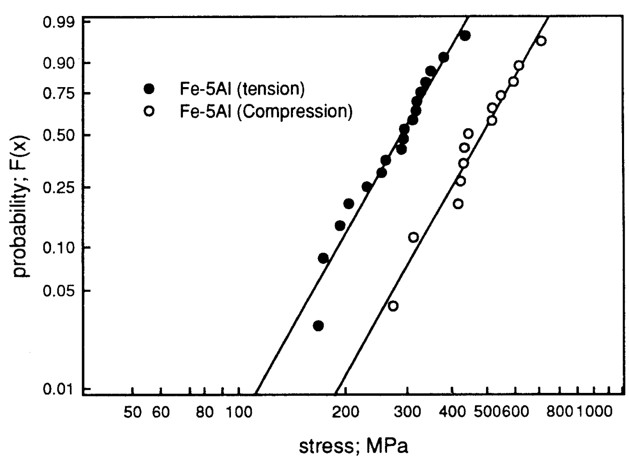
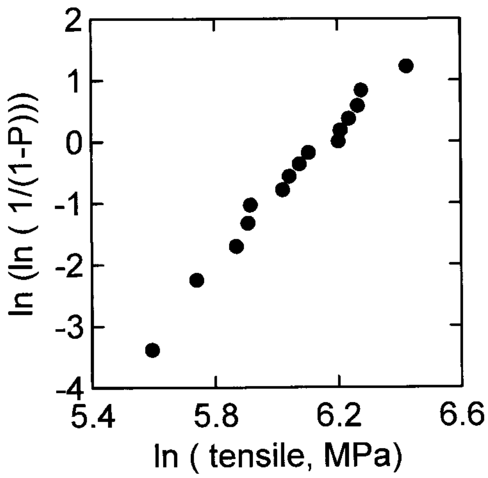


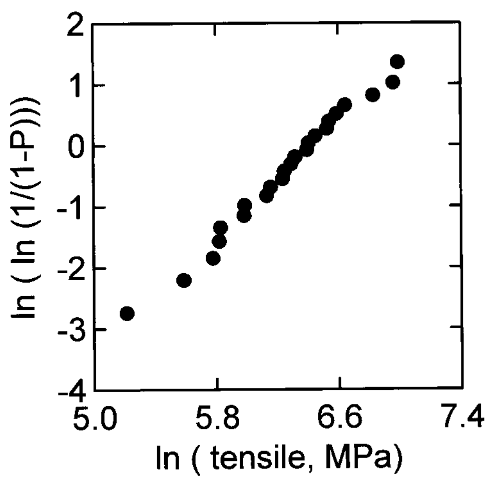
Fig. 5 Weibull cumulative distribution function (cdf) graph for Fe-5Al tensile and compression data

Originally, a three-parameter Weibull fit for all the data was made by adjusting the lower distribution limit to values between zero and the lowest measured value until the best fit was obtained (see Appendix). For the density distribution of all the compositions and for the density, hardness, and grain size for the Fe-5Al, the three-parameter Weibull distribution was the more appropriate analytical tool. However, for all the tensile strength distributions there was no difference between the two-parameter Weibull distribution and the three-parameter Weibull distribution, for which the minimal strength of the three-parameter Weibull distribution was zero. A two-parameter Weibull distribution implies that the tensile strength of a sample could be zero. (Note: A number of Fe-5Al samples broke during machining, prior to tensile testing, as shown in Table 3.) For the mean and expected strength values (See Appendix for explanation and derivation of the Weibull location parameter, x_m), there was a significant improvement in the strength of the mechanically alloyed Fe-[Ar] and Fe-2Al compacts over Fe-(as rec) (Fig. 6).

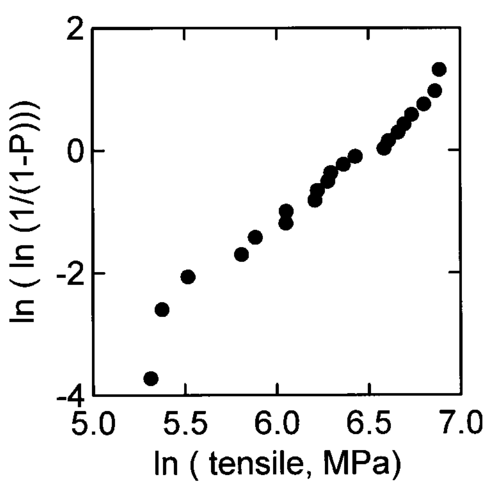
Weibull analysis also characterizes the distribution shape, and the Weibull shape parameter β(1) characterizes the amount of scatter on the data. The Gaussian distribution is graphically close to a Weibull cdf, which has a shape factor of approximately 3.44. For the data analyzed in this study, the Weibull shape parameters are quite distinct from 3.44, and thus, Weibull analysis is the more appropriate. Weibull shape factor analysis suggests that there is one strength distribution (or failure mechanism) for the Fe-5Al tensile and compression, another failure mechanism for the carbon alloys (Fe-2C and Fe-2Al-2C), and a third mechanism for the Fe-[Ar] and Fe-2Al alloys.



(a)



(b)



(c)

Fig. 6 Two-parameter Weibull distribution fits for hot-press tensile (a) Fe-(as received), (b) Fe-[Ar], and (c) Fe-2Al samples

4. Discussion

Hot-pressed attrition-milled iron-base nanostructured powders resulted in near full-dense compacts but with significant grain growth. In this study, five different iron powder compositions were attrition milled for 150 h resulting in almost identical powder characteristics. These powders, along with as-received iron powder, were hot-pressed at temperatures from 700 to 900 °C for 1 to 100 min. The resulting powder compacts were characterized for density, hardness, and grain size. Regardless of processing conditions used, the compacts were close to full theoretical density (>98% except for the Fe-5Al composition that was >95%).

Compacts of the as-received iron powder had a measured strength nearly identical to that of large-grain iron casting at 437 MPa for hot-pressed compacts Fe-(as-rec) versus 429 MPa for annealed AISI 1022 (Ref 14). Compaction of nanostructured iron powders processed in argon, Fe-[Ar] also produced nearly full-dense compacts with an enhanced tensile strength of 540 MPa.

Comparing statistical analyses of data from multiple samples prepared under similar hot-press conditions and data from samples prepared using different temperature and time processing conditions suggest that regardless of compact processing conditions, hardness, grain size, and tensile strength data all have the same probability distribution. The hot pressing of samples under similar conditions resulted in nearly identical compact densities, hardnesses, and grain size. Hot pressing under varying temperatures and times resulted in little change in the standard deviations of densities, but it did result in a significant increase in hardness and grain size standard deviation.

Statistical analyses showed no correlation between the compact characterizing variables, tensile strength and density, hardness, and grain size, or between the tensile strength and the processing parameters, temperature and time.

Care must be taken in the characterization of the compact tensile strength. The commonly used Gaussian analysis may provide misleading results. Analysis of the strength distribution indicates the data are better fit to a Weibull (two-parameter) distribution than to a Gaussian distribution. Weibull also provides a definite lower limit below which no stress failure will occur, which may be important for engineering applications.

The maximum tensile strengths for the Fe-(as-rec), Fe-[Ar], and Fe-Al compacts are quite different, but the minimum values are more closely grouped. These results are consistent with the failure mechanism being due to the presence of an inherent internal-flaw size distribution. The internal flaw of maximum size characterizes brittle failure, which is the flaw of weakest strength. This underlying behavior is precisely that upon which the Weibull distribution is based. Consequently, the Weibull cdf is an excellent choice to represent the tensile failure data. The presence of flaws and the resulting flaw size distribution were independent of the processing conditions. The presence of flaws was not detected by measuring the density, hardness, or grain size of samples.

The difference in average tensile strength of the Fe-(as-rec), Fe-[Ar], and Fe-Al compacts is due to a significant increase in the range of the maximum tensile strengths. Weibull analyses suggest the minimum tensile strength below which no failure would occur for these three compositions, and in fact for all compositions, was zero. The lack of correlation between tensile strength and density, hardness, and grain size suggests the limits in tensile strength may be due to the presence of internal flaws. There is a minimum flaw size that, if present, will result in immediate failure. The presence of flaws in powder compacts is inherent in the hot-press process and is not a function of the processing temperature or time. The presence of strength-controlling flaws in the compacts prior to testing was not detected by measuring density or hardness, or by optical examination. The increase in compression failure strength over tensile strength for the Fe-5Al is consistent with internal flaws being responsible for failure. In compression testing, small flaws are readily closed and would not contribute to failure, whereas larger flaws would be more difficult to close and could cause failure.

This study suggests that nanostructure characterizations of the milled powder, as well as many mesostructure characterizations of nanostructured compacts, may have limited applications in trying to characterize nanostructured compact strength. During compaction of the nanostructured particles, new material phenomena evolve, such as crack in the compact resulting from incomplete particle coalescence, which control some macroscopic properties such as strength.

5. Conclusions

- Hot pressing of nanostructured materials can produce compacts with very reproducible properties: density ±1.6% standard deviation, hardness ±4% standard deviation, and grain size ±4% standard deviation.
- However, tensile strength has a standard deviation in excess of 25%. Tensile strength of nearly full-dense compacts was unrelated to density, hardness, or grain size or to processing parameters of temperature and time; it appears to be related to inherent flaw distribution.
- Evaluation of strength and strength variability or distribution of hot-pressed compacts made from attrition-milled nanostructured powders suggests that the more informative statistical technique is Weibull analysis, which showed that the strength distribution starts at zero; that is, there is no minimal design stress.
- While the optimal tensile performance of these compacts made from nanostructured powders is promising, the large degree of uncertainty in the tensile strength, the characterization of the failure mechanism resulting from inherent flaw distribution, the inability to detect presence of these flaws, and tensile strength reached with little ductility may greatly limit their engineering applicability.

Appendix: Weibull Statistics

It is very easy to find the best Gaussian (normal) cumulative distribution function (cdf) for a set of data, which may be the primary reason it is so widely used for statistical failure analyses. Nevertheless, ease in statistical analysis does not imply a suitable cdf for the data. Consequently, the Weibull cdf also was considered in this study. In fact, the Weibull cdf was popularized because of its applicability for statistical analysis of materials behavior (Ref 15-18). The primary reason for this is that the Weibull cdf is the only nonnegative cdf that characterizes the behavior of the minimum of a collection of random variables. Hence, any process that can be characterized by the dominant flaw being the weakest element that causes failure will be the best represented by Weibull cdf (Ref 19). Statistical fracture analysis, especially brittle behavior as present with the materials tested in this study, is well represented by the Weibull cdf because the primary failure mechanism is dominated by the most severe flaw. Weibull cdf has been used for some time to characterize behavior of high-strength brittle materials (Ref 20-23).

Weibull analysis allows one to characterize data that come from a statistical distribution that has values from zero to infinity. The Weibull cdf assumes that there is a lower boundary on the data below which no data value is possible. For example, density, hardness, grain size, and tensile strength all have to be positive. In addition, there are circumstances where the minimum data value is not zero but has some finite value. For example, the density of a solid cannot be zero, or from experimental studies, the tensile strength of a material has been found always to be above some finite value. Weibull analyses provide a technique to evaluate this lower limit. Because the Weibull cdf has a lower limit, engineers can then design to these limits with a specified degree of certainty. The Weibull cdf is a very common distribution used to evaluate tensile properties of brittle materials and powder compacts where catastrophic failure often comes with no warning (Ref 22-24).

Weibull statistics often are referred to as “weakest link” analyses. The weakest link premise suggests that the system contains flaws, and that there is a flaw size such that when the load reaches a certain value, this flaw (if present) will cause failure (Ref 24, 25). During hot pressing, the possibility exists that regions contain bonding flaws. The distribution of bonding flaws and of flaw sizes is not known. But there is a greater likelihood that a flaw of a certain critical size will exist in any given volume of material. As the size of the volume increases, so does the likelihood of the critical flaw being present.

In its most general form, the Weibull distribution requires three parameters to be fitted to the data:

$$P_f(x) = 1 - \{\exp[(x - x_o)/x_m]^\eta\} \qquad \text{(Eq 1)}$$

where $P_f(x)$ = Weibull probability distribution for variable x, x = random variable (tensile stress, density, etc.), x_o = origin of the distribution (for a two-parameter Weibull this value is zero), x_m = characteristic life, and η = shape factor. The two-parameter Weibull distribution (where x_o is assumed equal to zero) can be fitted by a number of techniques.

In this study, three different statistical analysis methods were used to generate the Weibull distribution coefficients. The first method was the maximum likelihood method, which was tested by the Kolmogorov-Smirnoff statistical goodness of fit test (Table 6), to determine the Weibull coefficients. The second method used commercial statistical computer software (SYSTAT version 7.0 Survival package, HALLoGRAM Publishing, Aurora, CO) that generated two goodness-of-fit values: p-value (the closer to 1.0, the better the fit) and the initial regression score (the closer to 0, the better the fit). The third fit the data to a line (description following) and used the R^2 as the goodness-of-fit criteria (the closer to 1.0, the better the fit). All three techniques generated statistics for a two-parameter Weibull distribution.

The latter technique (which is less accurate than the maximum likelihood method (MLE) analysis used to determine the Weibull statistics in the main text) was used to generate Fig. 6 and 7. The Weibull statistics were determined by first ordering the (tensile) data, x_i, from low to high, and then assigning a ranking to the individual data. This ranking was then transformed to a weighted cdf, P_i = {1 – (ranking of the ith tensile data point – 0.5)/total number of data points}. The Weibull parameters are then determined by transforming Eq 1 to a linear equation with the dependent variables, ln (ln [$1/P_i$]), and the independent variable, ln (x_i). The transformed data plotted and fitted using linear regression analysis (Ref 22) using the equation:

$$y_i = a + b \cdot x_i \qquad \text{(Eq 2)}$$

where y_i = ln (ln{[1/{1 – (rank of ith data point –0.5)/total number of data points]}),

x_i = ln (ith tensile value).

The coefficients in equation in Eq 2 are related to the constants in the two-parameter Weibull cdf by $a = \eta \cdot \ln(x_m)$ and $b = \eta$. The closer the data fit to the linear approximation, R^2, the better the determination of the Weibull coefficients and the better the data fit a Weibull distribution.

The parameters in the Weibull cdf are related to physical quantities in the following way: η is characteristic of the scatter and x_m is approximately the mean value of the data as represented by the Weibull cdf. Thus, η reflects the reproducibility of the statistical outcomes and is measured by the slope, b, of the line (Eq 2). As the slope increases, the scatter in the data decreases. Whereas, x_m primarily reflects the typical behavior being measured. The Weibull cdf is quite robust in that it can adequately characterize a variety of statistical behaviors.

The three-parameter Weibull can be determined from multiple applications of the two-parameter analysis described above. Instead of setting x_o equal to zero, the origin offset can be subtracted from the original data and the two-parameter analysis described above can be applied to this new set of data, that is, the expression ($x - x_0$) in Eq. 1 can be combined into a new variable, x'. This process can be repeated until the optimal fit of the data is accomplished. See Table 7 for determination of the three-parameter fit for an example where the distribution did not start at zero—Fe-5Al density, and another where the distribution does start at zero—Fe-[Ar] tensile.

Table 7 Three-parameter Weibull analysis determined from two-parameter Weibull analysis by using a nonzero offset for the determinate variable, *x*

Offset from zero, x'	Shape factor, η	p-value	Regression score	R^2 correlation
Fe-5Al at density g/cm^3 <5.55>±0.09, minimum measured value 5.36 g/cm^3				
0	73.7	0.594	1.04	0.924
3.0	33.7	0.632	0.91	0.929
4.0	20.4	0.680	0.77	0.934
4.5	13.9	0.741	0.60	0.949
5.0	6.91	0.890	0.23	0.952
5.2(a)	4.12	0.892	0.23	0.956
5.3	2.60	0.515	1.33	0.937
5.34	1.85	0.168	3.56	0.888
Fe-[Ar] at tensile strength <287>±73, minimum measured value 168 MPa				
0(a)	2.42	0.829	0.373	0.990
25	2.30	0.757	0.556	0.989
50	2.17	0.646	0.874	0.985
75	2.03	0.487	1.439	0.976
100	1.86	0.281	2.540	0.946

This is accomplished by assuming there is a minimum value, $\mathbf{x}_0$, below which there is zero probability that x exists. This value is then subtracted from x, forming a new variable: $x' = (x - x_0)$. The two parameter Weibull analysis is now conducted on this new variable. X' is varied until the linear regression determines the best fit to the data. Best statistical fit can be determined by maximizing the p-value or the R^2 value or minimizing the regression score. (a) Best fit

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