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Properties of Extruded Disintegrable Metal Composites

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Abstract

Recently, a lightweight, disintegrable material with high strength and high ductility has been successfully used in oilfield tool applications to save well operation time and cost. The material is a powder metallurgy composite consisting of a metal matrix and mechanical/chemical reinforcements. Warm extrusion was used to produce rods and tubes of the material from cold pressed green preform. Disintegration rates of the extruded materials with different compositions range from 0 – 300 mg/cm^2/hr in 3% KCl at 93.3 ^{o}C with a strength of > 60 ksi (414 MPa) and ductility of 7 – 15%. This paper presents the microstructure, disintegration, and mechanical properties of the extruded composite. The results prove that disintegration properties can be controlled by altering the chemical composition of the reinforcements while maintaining good strength and ductility.

Introduction

Disintegrable metal matrix composite, DMMC, is a unique powder metallurgy composite where a base powder material is coated or modified via milling/CVD with customizable metallic or ceramic chemistries. Nano/micron level chemistries and thickness ultimately decide the balance between material strength and its dissolution in different wellbore fluids. The ultimate goal of this material development is to provide a disintegrable, high strength material system that can be used in interventionless downhole tools for completing different formation conditions. Completion devices such as fracturing balls, discs, plugs, and perforating gun bodies are often used for downhole fluid and pressure control.

Current polymer material must be milled away, flowed back or otherwise removed before production. Severe deformation of currently used materials that prevent flowback have been reported, requiring costly intervention operations to either remove or replace the tools and resulting in higher operational inefficiency. Using DMMC eliminates this possibility. The material is inherently designed for in-situ disintegration in downhole environments at a controlled customizable rate.

Conventional degradable materials have a well-known tradeoff between disintegration rate and mechanical strength, which limits their utility. The need for a high-strength disintegradable system is desirable; however, no system can provide the strength needed for this application [1 - 4]. An increased dissolution rate often lowers the mechanical strength of the material below an acceptable threshold for many downhole applications. DMMC provides the option to set the corrosion rate while maintaining its structural integrity in wellbore conditions.

Extrusion Process of DMMC

Extrusion is necessary to create a cost effective and post processing alternative without losing mechanical and disintegration properties. This can be accomplished by extruding green preform billets without losing the unique material properties of DMMC and creating objects of a fixed cross-sectional profile. The two advantages of this process are: 1. the production rate can be increased by extruding large preform billets 2. Higher strength can be achieved through work hardening of the material.

The press used for extrusion is illustrated in **Fig. 1**. A pre-heated preform billet, **Fig. 2**, is loaded into a container and pressed through a hot die. The starting billets were 4 in. (10.2 cm) and 9 in. (22.9 cm) OD and pushed through various tube and rod dies.

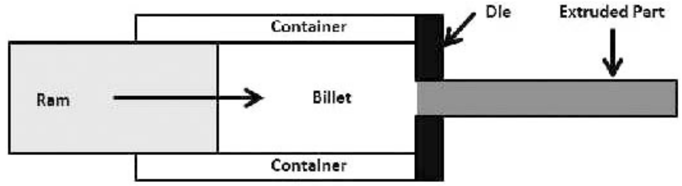

Fig. 1. Extrusion press diagram used for DMMC extrusion.

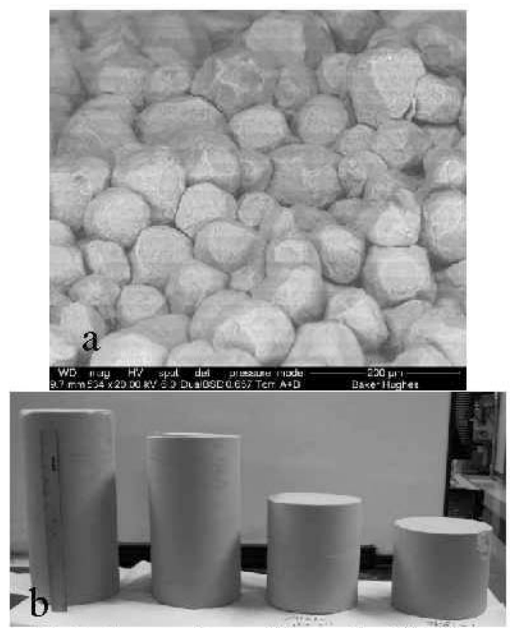

Fig. 2. Green preform of (a) powder, (b) billets.

Extrusion involved using critical pre-heating and process temperature controls to maximize results. The billets, dies, and containers were pre-heated to elevated temperatures for processing DMMC to the shape desired. Once the billet reached the optimum temperature, it was transferred to a container and pressed through the die with ram pressure. The ram pressure exerts pressure on the body which is moving through a fluid medium. The drag force exerted on the body if defined as (1), where P is the pressure, ρ is the density of the media, and v is the velocity of the body.

$$P = \rho v^2 \tag{1}$$

Table I lists the materials extruded at various reduction ratios. Each material family is composed of nanostructured coating or reinforcement materials.

Table I. DMMC materials extruded through various rod and tube dies.

ID	Starting Billet OD, cm	Final OD (ID), cm	Ratio
S1	10.80	2.54	18.1
S2	10.80	5.08	4.5
S3	20.32	7.62	7.1
S4	20.32	10.01	4.1
S5	20.32	7.62 (6.35)	23.7
S6	20.32	10.01	4.1
S7	20.32	12.07	2.8
S8	20.32	8.89 (6.35)	10.9
S9	20.32	13.34 (10.80)	6.9

The extrusion ratio is the cross-sectional area of the billet divided by the cross-sectional area of the final extruded part:

$$\mathrm{ER} = \frac{A}{A'} \tag{2}$$

Extruded Parts

DMMC materials were extruded into different forms, including solid rods and tubes. Photos of extruded S5 and S8 tubes are shown in **Fig. 3**. The original billet was 8 in. (20.32 cm) OD with final tube OD of 3 in. (7.62 cm) (a) and 3.5 in. (8.89 cm) (b) with 0.25 in. (0.635 cm) and 0.5 in. (1.27 cm) wall thickness, respectively. **Fig. 4** shows both a 5.25 in. (13.34 cm) OD tube with 0.5 in. (1.27 cm) wall thickness (a), and 4.75 in. (12.07 cm) OD extruded rod (b).

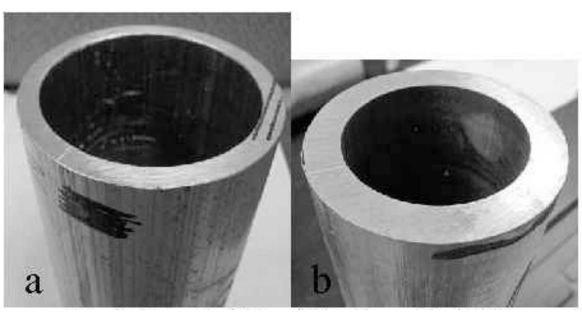

Fig. 3. Extruded tubes (a) 7.62 cm OD, 0.635 cm wall thickness, (b) 8.89 cm OD, 1.27 cm wall thickness

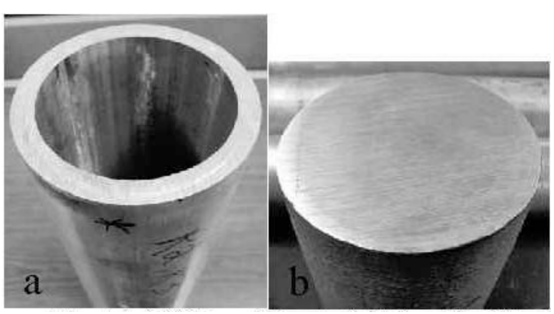

Fig. 4. (a) 13.34 cm OD extruded tube with 1.27 cm wall thickness, (b) 12.07 cm OD extruded rod.

Extruded rods of 1 in. (2.54 cm) OD were also produced (see **Fig. 5**) from a 4.25 in. (10.80 cm) OD starting billet. Test coupons for disintegration rates and compressive strength testing were machined according to ASTM E9.

Fig. 5. 2.54 cm extruded rods.

Properties and Microstructure of Extruded DMMC

Disintegration, mechanical, and microstructure properties provide the unique characteristics of extruding DMMC. Billet pre-heating temperature, extrusion rate, and die temperature play significant roles in providing the required properties needed for downhole use. Improper heating can drastically alter overall DMMC properties. To test these properties, 0.5 in. (1.27 cm) OD x 1.0 in. (2.54 cm) L coupons were machined from the final extruded parts and tested for the previously mentioned properties.

Disintegration properties

Rate of disintegration (ROD) testing was completed using a dynamic test cell in the presence of 3% KCl at 93.3°C. The sample was fully submerged in brine for a specific amount of time, and periodically checked for dimensional change and weight. The results are presented in Table II as mg/cm^2/hr, where the difference in weight is divided by the difference in area and total testing time:

$$\mathrm{ROD} = \frac{\Delta wt}{\Delta A \times t} \tag{3}$$

Table II. Disintegration rates of various extruded rods and tubes.

ID	ROD (mg/cm^2/hr)
S1	0.649
S2	0.37
S3	2.24
S4	2.76
S5	6.365
S6	1.33
S7	58.78
S8	21.09
S9	15.12

Disintegration rates can be controlled using reinforcement and coating chemistry while maintaining uniform disintegration, as shown in **Fig. 6 (b).** In one case, rates as high as 300 mg/cm^2/hr were achieved. The tested coupons appeared dark grey with surface pitting when compared to **Fig. 6 (a)**.

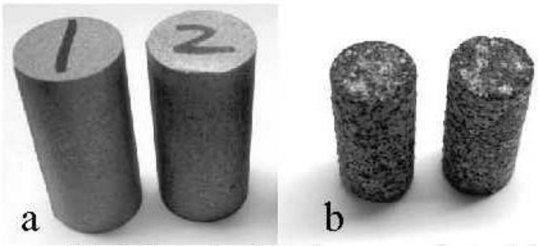

Fig. 6. Example of sample coupons of extruded parts (a) prior to testing and (b) after exposure to brine at 93.3°C .

Mechanical testing. The extruded product was tested for mechanical properties as shown in Table III (use table I as reference to extrusion type). 2.54 cm OD (S1) rods averaged 67.5 ksi (465 MPa) UCS with 7.1% strain while the 5.08 cm OD (S2) rod resulted in 63 ksi (434 MPa) UCS and 6.9% strain. The slight difference in UCS can be attributed to the lower extrusion ratio shown in table I, 18.1 versus 4.5.

Likewise, S3 and S4 billets were extruded into 7.62 cm and 10.01 cm OD rods, each showed average UCS of 62 ksi (428 MPa) with 11.4% and 13.3% strain, respectively. In this case, the slight difference in ratio, 7.1 versus 4.1, didn't affect the UCS.

Table III. Mechanical data of various DMMC rods and tubes.

ID	UCS, KSI (Mpa)	Strain % at Max UCS
S1	67.45 (465)	7.12
S2	63 (434)	6.93
S3	62.3 (430)	11.42
S4	62 (428)	13.32
S5	69.1 (476)	13.25
S6	53.7 (370)	13.23
S7	63.2 (436)	13.84
S8	54.8 (378)	10.76
S9	61.8 (426)	13.98

The ultimate compression strength (UCS) was plotted against the disintegration rate for rods and tubes (**Fig. 7** and **Fig. 8**, respectively). The disintegration can be controlled for a given strength.

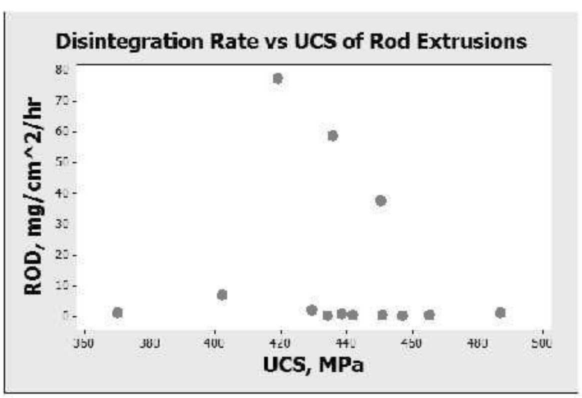

Fig. 7. Disintegration rate vs. rod UCS.

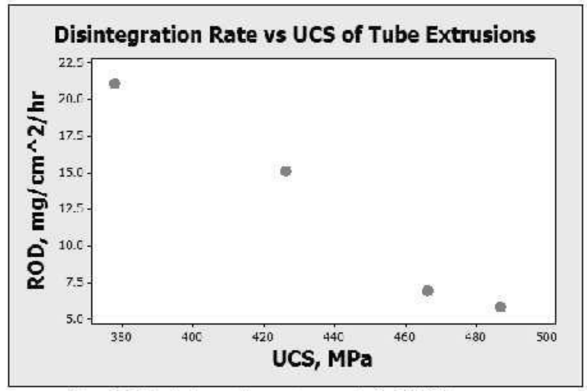

Fig. 8. Disintegration rate vs. tube UCS.

In **Fig. 9**, the UCS is plotted versus the extrusion ratio. The chart shows rods as solid dots and tubes are triangles. The UCS and extrusion ratios are proportional. Increased strength can be achieved with higher extrusion ratios.

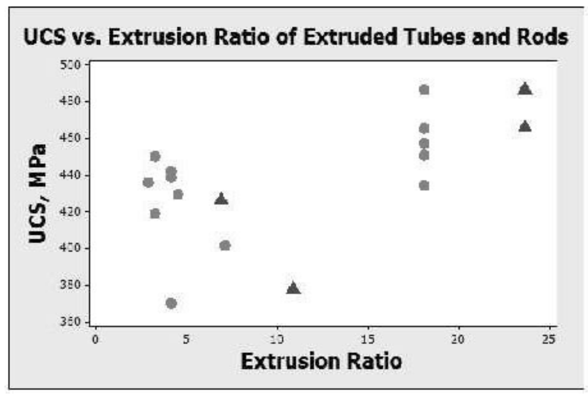

Fig. 9. UCS and extrusion ratio of extruded rods and tubes.

Microstructures.
As listed in Table I, rods and tubes were extruded into various ODs/IDs from forged and CIP billets. The microstructure of forged S2, **Fig. 10**, has a uniform structure, and the individual grain boundaries are clearly visible around the base particle. Once extruded, **Fig. 11**, the grain boundary, is spread along the direction of extrusion, and is depicted by the arrow.

Fig. 12 shows a micrograph image of as-extruded S4 extruded from a preform billet, as in **Fig. 2**. The extrusion direction is denoted with the arrow, but has a less visible grain boundary. The boundary difference is because of the pre-manufacturing conditions chosen for each powder. All materials extruded exhibited similar microstructure profiles relative to their prepping conditions. The increased surface area is clearly visible in the extruded profiles, and contributes to a slight increase in the disintegration rate, compared to as-forged.

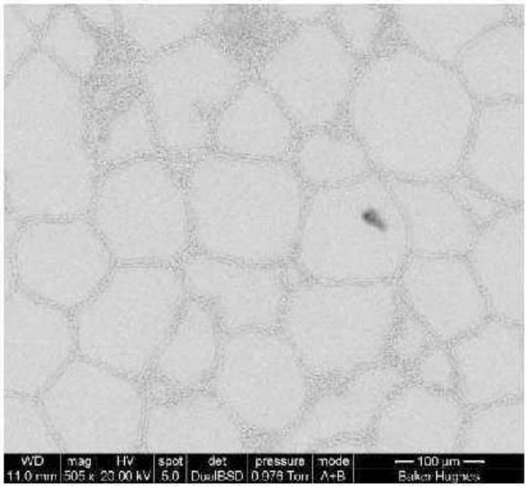

Fig. 10. SEM cross-section of forged S2.

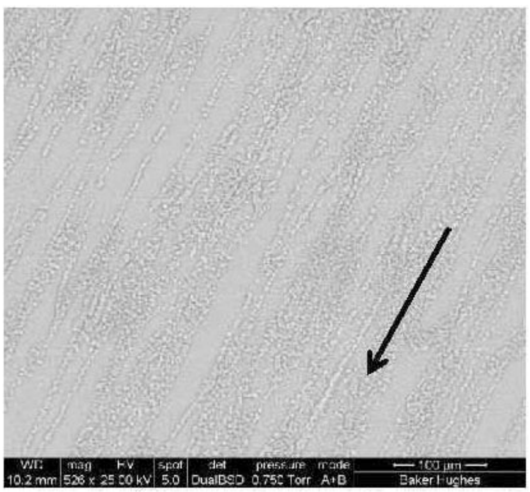

Fig. 11. SEM image of extruded S2. Direction of extrusion is depicted by arrow.

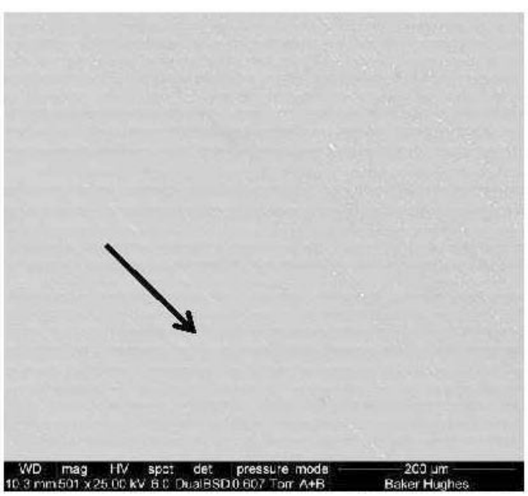

Fig. 12. SEM image of extruded S4. Direction of extrusion is depicted by arrow.

Conclusion

Disintegrable metal matrix composites with unique properties and microstructures are ideal structural materials for making interventionless downhole tools or components. Extrusion of preform DMMC billets, to tubes and rods, has been successfully completed. Extrusion from the preform billet shows we can achieve ROD and UCS comparable to as-forged. Disintegration rates can be controlled from 0 – 300 mg/cm^2/hr, in 3% KCl at 93.3 °C while maintaining >60 ksi (414 MPa) strength.

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