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# Microstructure control of cold-sprayed pure iron coatings formed using mechanically milled powder



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# ABSTRACT

Nanocrystalline metallic materials with grain sizes of less than 100 nm exhibit high strength and unique characteristics, such as high levels of ductility and corrosion resistance. Mechanical milling techniques can be used to produce large amounts of nanocrystalline metallic powder. However, it is essential to perform a sintering treatment, which induces coarsening of the crystal grains, to form a bulk material from the mechanically milled powder. The cold spray technique is expected to effectively form a nanocrystalline metallic coating using the mechanically milled powder. This study focuses on pure iron coating, which is formed by a cold spray technique using mechanically milled pure iron powder. Pure iron powder, with crystal grains of ~100 nm, was obtained by a mechanical milling process, and was deposited onto a low-carbon steel substrate by cold spraying. It was confirmed that a dense, nanocrystalline coating with a hardness of over 300 HV can be achieved; however, the deposition efficiency of this coating was half of that of the coating formed using the as-received powder. The improvement of the deposition efficiency and properties of such coatings was attempted using powder mixtures comprising both milled and as-received powders. The results indicate that the deposition efficiency and microstructure, directly linked to the mechanical properties of the coating, can be controlled by varying the content of the as-received powder of the mixture.

### 1. Introduction

In power and chemical plants, high chromium steels or stainless steels, which exhibit high strength and high corrosion resistance, are employed for components, such as pipes and turbine blades. Typically, these excellent properties are achieved by adding rare metals or strategic materials to the base metal. However, for the effective utilization of resources, the improvement of these properties without the use of rare metals or strategic materials is desirable.

Grain refinement is a technique used for the reinforcement of metallic materials without the addition of rare metals or strategic materials. In particular, nanocrystalline metallic materials, which are generally defined as materials with grain sizes smaller than 100 nm, have high strengths exceeding those of coarse-grained and even alloyed metallic materials [1]. Furthermore, nanocrystalline metallic materials can exhibit high levels of ductility and corrosion resistance [2–4]. These unique characteristics have attracted the attention of several researchers. A number of techniques have been proposed to achieve nanocrystallization of metallic materials, such as thermo-mechanicalcontrolled processes, equal-channel angular pressing, accumulative roll-bonding, and mechanical-milling [5-8]. These techniques are based on the introduction of severe plastic strain to metallic materials and dynamic recrystallization. Among these techniques, mechanical milling can be used to produce large quantities of nanocrystalline metallic powders relatively conveniently. During the mechanical milling process, the powder material is repeatedly deformed by the high-energy impact of the balls. However, it is essential to conduct a sintering treatment at high temperature to form a bulk material from the nanocrystallized powder produced by the mechanical milling process. Hightemperature treatments induce coarsening of the nanocrystal grains obtained by the mechanical milling process.

Cold spray techniques are based on the impact and deposition of numerous fine and solid particles accelerated by a supersonic gas flow [9,10]. Such cold spray processes are performed below the melting temperature of the powder material; thus, they are capable of maintaining the microstructure of the feedstock powder. This suggests that nanocrystalline metallic coatings can easily be achieved by the application of mechanical milling, combined with cold spraying. A number of nanocrystalline coatings, such as aluminum alloys, copper alloys, nickel alloys, and intermetallic alloys, formed by cold spraying using

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#### Table 1

Chemical composition of the pure iron powder [unit: wt%].

С	Si	Mn	Р	S	Fe
0.007	0.01	0.06	0.015	0.006	Bal.

mechanically milled powder, have previously been investigated [11–16]. Ajdelsztajn et al. reported that an Al 5083 powder with nanocrystalline grains in the range of 20–30 nm can be obtained by performing mechanical milling under a liquid nitrogen environment. In addition, it has been confirmed that nanocrystalline Al 5083 coatings can be formed by cold spraying; the hardness values of these coatings are significantly higher than those of cast or cold worked Al 5083 [17]. Kumar et al. confirmed the successful formulation and deposition of nanocrystalline Ni–20Cr powder, with an average crystal grain-size of 10 nm, onto SA 516 boiler steel by a cold spray technique [18,19]. The nanocrystalline Ni–20Cr coating provided superior high-temperature oxidation resistance and erosion–corrosion resistance compared to those of boiler steel and coarse-grained Ni–20Cr coatings.

As shown by these reports, nanocrystalline metallic coatings can be obtained by employing mechanical milling combined with cold spraying. This study focuses on a pure iron coating, formed by cold spraying using mechanically milled feedstock powder, as an alternative material to high chromium or stainless steels. Commercially available pure iron powder was mechanically milled under various conditions, and characterized to determine the optimal milling conditions. The powder was milled under specific conditions, and then, it was deposited onto a low-carbon steel substrate by cold spraying. The deposition efficiency, microstructure, and hardness of the obtained coating were evaluated using scanning electron microscopy (SEM), electron backscatter diffraction (EBSD) analysis, and hardness testing.

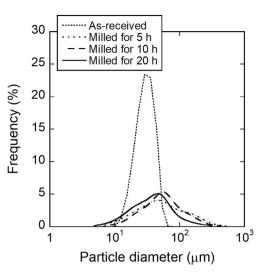


Fig. 2. Particle-size distribution of the as-received powder and the powders that were milled for various periods.

#### 2. Experimental procedures

## 2.1. Mechanical milling conditions

A commercially available pure iron powder (ITOH KIKOH CO., LTD., Japan) was employed as the feedstock powder for the cold spray process. The chemical composition of the iron powder, shown in Table 1, was identified using an optical emission spectrometer (PDA-5500, Shimadzu Corporation, Japan) and a carbon and sulfur analyzer (EMIA-220V, HORIBA, Ltd., Japan). The pure iron powder was treated by using a commercial planetary ball milling equipment (Planetary Micro Mill PULVERISETTE 7, Fritsch Co., Ltd., Germany). The iron

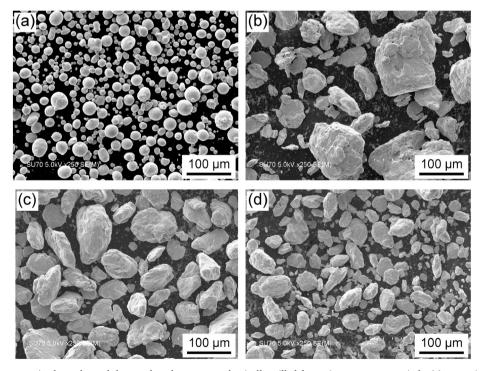


Fig. 1. SEM images of the as-received powder and the powders that were mechanically milled for various treatment periods; (a) as-received, (b) milled for 5 h, (c) milled for 10 h, and (d) milled for 20 h.

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