



# Development of nano-crystalline cold sprayed Ni–20Cr coatings for high temperature oxidation resistance



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## ABSTRACT

In the current investigation a pre-synthesized nano-crystalline Ni–20Cr powder was deposited by cold-spray technique on SA 516 steel. The powder was synthesized by ball milling approach. The nano-crystallinity of the developed coating was established by crystallite size measurements from XRD profile, which was further endorsed by TEM analysis. High-temperature oxidation behavior of uncoated and coated samples was studied under cyclic isothermal conditions at 900 °C for 50 cycles in a laboratory tube furnace. The oxidation rates for the bare and coated steel were evaluated in terms of weight gain data. Different characterization techniques, such as X-ray diffraction (XRD), scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS), and transmission electron microscopy (TEM) analyses were utilized to characterize the oxide scale. The hardness and oxidation resistance of the steel increased after the application of coating. The reduction in oxidation rate of the base steel was 94%. Moreover, the developed nanostructured coating was observed to reduce weight gain by 64% in comparison with micron-sized Ni–20Cr cold spray coating thus offers a higher oxidation resistance. This may be attributed to relatively denser structure, lower porosity and lower oxide content of the nanostructured coating.

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## 1. Introduction

High-temperature degradation of heat-transfer pipes such as super heater tubes in coal-fired boilers is a serious concern. Continuous thinning of the boiler tubes in the working environment due to erosion–corrosion (E–C) leads to premature rupture of boiler tubes. This leads to increase in downtime of the boilers and hence causes tremendous economic losses. Keeping in view of these facts, several methods to counter this degradation have been suggested and investigated. One possible way to counter this surface degradation of boiler tubes is application of thermal spray coatings. Thermal spraying has gained a wider popularity worldwide due to its versatility and several other advantages such as production of high-quality coatings, high production rates, wide range of coating compositions and thicknesses, minimum thermal degradation of the substrate materials, on-site applications and low promising costs [1–4]. Thermal spray coatings can induce tailor made surface properties without affecting the metallurgical properties of the substrate material and without any significant damage to environment, in comparison to their counterparts [5–8]. In thermal

spraying, the coating material is heated rapidly in a hot gaseous medium, and simultaneously propelled at a high velocity onto a prepared substrate surface, where it is deposited to produce the desired thickness of coating, by passing a preset number of passes of a spraying gun [9,10]. However during thermal spraying the chances of oxidation of feedstock powder are always there [5–8,11,12], therefore one of the recent variants of thermal spraying, namely cold-spray spraying (CS), was developed at the Institute of Theoretical and Applied Mechanics of the Siberian Division of the Russian Academy of Science (ITAM of RAS) [13–15]. Since, CS is a solid state process, it may be a promising thermal spray process to develop nanostructured coatings with low oxidation content.

Nanostructured coatings, which contain grains or clusters below 100 nm, or layers or filaments of that dimension [16], have attracted the attention of many researchers worldwide due to their excellent properties in comparison to micro-structured conventional coatings, such as hardness, ductility, corrosion and wear resistance [17–19]. It is learnt that mechanical milling of powders is one of the approaches to synthesize nanostructured powders [20,21]. In a typical ball milling process, the powder particles are subjected to repeated impacts, which give rise to cold welding of these particles [17]. With subsequent impacts, these welded particles get fractured. This process of welding

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**Table 1**  
Composition of the Ni–20Cr coating powder (wt.%) for the cold-sprayed coating.

Type of powder	Blended powders (wt.%)		
	Micron-sized	Nano-sized	Nano-sized
Name of powder	Ni (CA <sup>a</sup> ) {P1}	Ni (BM <sup>a</sup> ) {P2}	Cr (BM <sup>a</sup> ) {P3}
Composition (%)	64%	16%	20%
Particle size	74 $\mu\text{m}$	67 nm	60 nm

<sup>a</sup> CA—commercially available, BM—ball milled.

**Table 2**  
Parameters used for ball milling of the Ni–20Cr powder.

Parameter	Value
Ball-to-powder (B:P) weight ratio	10:1
RPM	300
Running time (min):pause time (min)	30:10
Process control agent	Toluene

**Table 3**  
Process parameters for the cold-spraying of Ni–20Cr powder on SA 516 steel.

Process gas	Air
Gun temperature	450 °C
Gun pressure	19 bar
Process gas flow rate	1.96 m <sup>3</sup> /min
Powder feed rate	113 g/min
Carrier gas	Air
Flow rate of gas	1.96 m <sup>3</sup> /min

and fracturing keeps on repeating itself. This leads to refinement of the grain size, and finally forming nano-crystalline microstructure. Moreover, cold spraying can effectively be used to deposit nanostructured powders, since low process temperatures help to retain nano-crystallinity of feed stock powder.

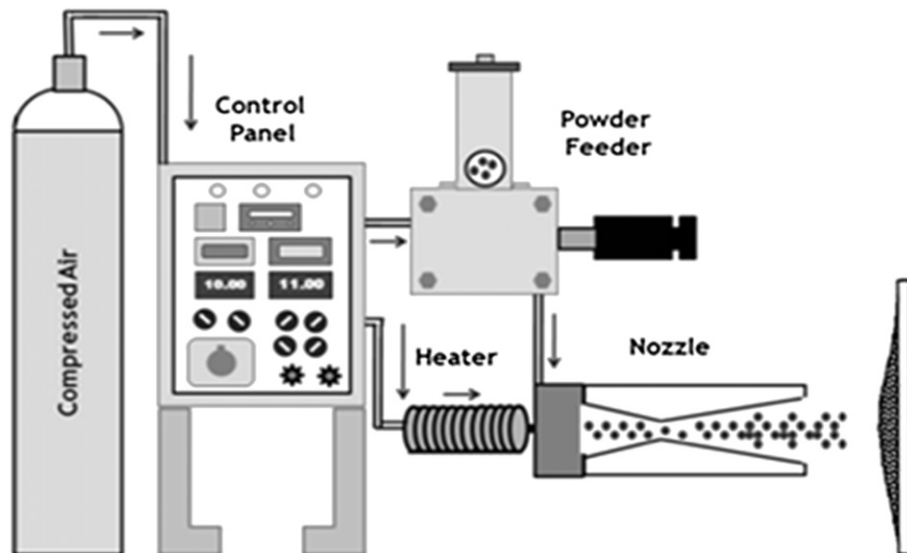
Nickel-based alloys possess several attractive properties such as wear, erosion–corrosion resistance, and good thermal conductivity. Due to these properties, nickel based coatings are frequently considered for the prevention of erosion–corrosion of boiler tubes [22–24]. Further, Ni–Cr nanostructured coatings are found to exhibit higher corrosion

resistance owing to their relatively low porosity and high grain boundary area. These attributes help to enhance the diffusion along grain boundaries thereby resulting into the formation of dense chromia-rich oxide scale in these nanostructured coatings. The authors have already reported some work [23], in which the same in-house developed Ni–20Cr crystalline powder was used to develop high temperature resistant coating on this steel by high-velocity oxy-fuel spraying. Subsequently the high temperature oxidation behavior of this coating has been evaluated with an aim to establish its usefulness for high-temperature applications such as boiler tubes. The presented work is a small portion of a bigger research program. The authors have also evaluated the erosion–corrosion behavior of these coatings in an actual boiler environment. The aim of the cyclic oxidation testing is to have a basic understanding of the high temperature oxidation behavior of the coating. This will provide the information about growth of the oxide scale and spallation tendency of scale, as well as, coating under accelerated thermal cyclic loading. In the current work the CS technique has been utilized to develop the coatings of this nano-crystalline powder on SA 516: Grade 70 (hereafter denoted as SA 516) steel.

## 2. Experimental details

### 2.1. Substrate and coating material

The substrate steel selected in the present study was SA 516 boiler steel: commonly used boiler tube material. This steel is widely used in fabricating boiler tubes in power plants of Northern India. The chemical composition (wt.%) of steel is carbon (C) 0.27; manganese (Mn) 0.93; silicon (Si) 0.1; sulfur (S) 0.06; phosphorous (P) 0.05 and balance iron (Fe). The test specimens of length: 20 mm; width: 15 mm; and thickness: 5 mm dimensions were prepared from the steel. These specimens were polished using emery papers of 220, 400, 600, 1000, and 1200 grit followed by fine polishing with emery papers of 1/0, 2/0, 3/0 and 4/0 grades. Subsequently, the specimens were polished down to mirror finish on a cloth wheel polishing machine using an alumina powder suspension as part of the standard pre-spray process for specimen preparation. The nano-crystalline Ni–20Cr powder was synthesized by blending three types of powders in a planetary ball mill. One of the powders was a commercially available Ni-powder (LobaChemie, India) having 99.9% purity and 74  $\mu\text{m}$  particle size, whereas the other powder constituted of pre-synthesized Ni nano-particles (ball-milled) having an average particle of 67 nm (vol.%). The third starting powder comprised pre-synthesized Cr nano-particles (ball-milled) with an



**Fig. 1.** A schematic of the cold spray coating process.

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