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In-situ observations of single micro-particle impact bonding

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ABSTRACT

We study supersonic impact of individual metallic microparticles on metallic substrates, that is, the unit process of materials buildup in cold spray coatings/additive manufacturing. We resolve the moment of impact bonding through real-time observations of single particle impacts with micron-scale and nanosecond-level resolution. We offer the first in-situ observation of a material-dependent threshold velocity, above which the particle undergoes an impact-induced jet-like material ejection and adheres to the substrate. We report direct measurements of critical velocities for structural metals, which unlike in nozzle experiments, are not affected by process-related complexities obscuring particles' kinetic and thermal histories.

Understanding material behavior under high velocity impact is the key to addressing a variety of fundamental questions in areas ranging from geological cratering [1] to impact-induced phase transformations [2], spallation [3], wear [4], and ballistic penetration [5]. Recently, adhesion has emerged in this spectrum since it has been found that micrometer-sized metallic particles can bond to metallic substrates under supersonic-impact conditions [6–11]. The phenomenon of impact-induced adhesion of microparticles has led to the emergence of cold-spray coating, which has not only proved successful in making corrosion-, wear- and fatigue-resistant coatings [12–20], but also opened a new window in structural repair [21] and additive manufacturing [22].

In cold spray coating, researchers have repeatedly observed a "critical velocity", a threshold above which supersonic particles adhere to the substrate instead of rebounding [7,23–25]. A variety of mechanisms such as adiabatic shear instability [7], localized melting [26], viscous-type mechanical interlocking [27], interfacial restructuring or amorphization [28], and oxide-layer break-up [29] have been put forth to explain this phenomenon. Among these mechanisms, adiabatic shear instability has acquired more consensus [9,11,30–32] as it enjoys support from Lagrangian finite element simulations [7,23], but has not been directly observed. These mechanisms are not mutually exclusive. However, for a fixed particle/substrate materials pair and fixed processing conditions, the premise of a single critical velocity suggests that a single mechanism may dominate the binary separation between bonding and not. A cold spray deposit comprises millions of splats, each of which interacts with a carrier gas and other particles in a unique way. Each splat experiences distinct thermal and kinetic histories, impacts the deposit and is impacted by other particles in an unknown fashion. All these complexities give rise to a variety of phenomena that can potentially obscure the main phenomenon leading to bonding.

In our view, a systematic approach to focus on the unit process of material build up in cold spray, i.e., impact bonding of a single microparticle, without the complexities of nozzle experiments, is much needed for a clear understanding of bonding in cold spray. Furthermore, we attribute the lack of consensus on the operative mechanisms, in part, to a lack of real-time studies of supersonic micro-particle impact. Such studies require spatial (micron) and temporal (nanosecond) resolutions much finer than those provided by existing experimental techniques.

Here, we offer an alternative approach by conducting the first in-situ single-particle study of supersonic adhesion of metallic microparticles. The present letter is based on Ref. [33], and employs an in-house-designed microscale ballistic test platform to accelerate micrometer-size particles [34,35] and observe their impact behaviors in real time [36,37]. As schematically shown in Fig. 1, a laser excitation pulse is focused onto a launching pad assembly from which single metallic particles are launched toward a target sample by ablation of a gold layer and rapid expansion of an elastomeric polyurea film. The particle approach and impact on the target are observed in real time using a high-frame-rate camera and a synchronized quasi-cw laser imaging pulse for illumination. The launching pad assembly follows the design described by Lee et al. and Veysset et al. in refs [35,36]. 210-μm-thick glass substrates (Corning No. 2 microscope cover slip) were sputter-coated with a 60-nm thick gold film. A mixture of polycarbodiimide-modified diphenylmethane diisocyanate (Isonate 143 L MDI, Dow Chemicals) and oligomeric diamine (Versalink® P-650, Air Products) with a weight ratio of 1:3 was spin-coated on the gold-coated substrates at 750 RPM for 5 min to yield a film thickness of 30 μm after 24-hour curing at

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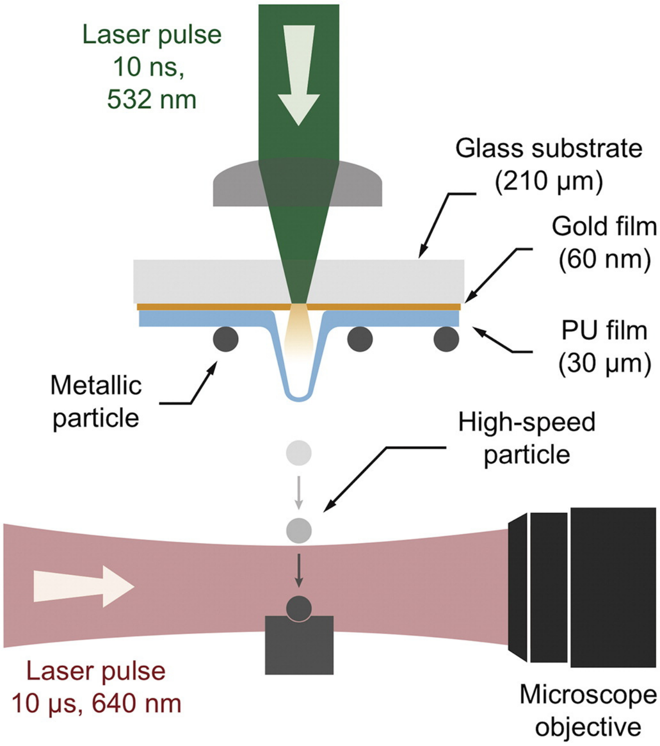

Fig. 1. Experimental platform for microparticle impact test and real-time imaging.

room temperature. Film thicknesses were measured using a 3D laser scanning confocal microscope (VK-X200 series, Keyence). Metallic particles were deposited on the substrates using lens cleaning papers to spread drops from a suspension of particles in ethanol.

For each experiment, a laser excitation pulse (pulsed Nd:YAG, 10-ns duration, 532- nm wavelength) was focused onto the launching pad assembly from where the metallic particles were ejected. Upon laser ablation of the gold film, particles were accelerated to speeds ranging from approximately 100 to 1200 m/s, controllable by adjusting the laser excitation pulse energy (from 2 up to 60 mJ). 16-image sequences showing impact were recorded with a high-frame-rate camera (SIMX 16, Specialised Imaging) using a laser pulse (30-μs duration, 640-nm wavelength SI-LUX640, Specialised Imaging) for illumination. The high-speed camera comprises 16 CCDs that can be triggered independently to record up to 16 images with exposure times as short as 3 ns.

We considered four structural metals that cover a wide range of relevant cold spray applications (Al, Cu, Ni and Zn), and conducted impact experiments with matched particle and substrate materials pairs. We purchased two batches of Al powder particles with nominal particle sizes of 20 and 31 μm from Valimet (Stockton, USA). We also purchased Cu, Ni, and Zn powder particles with nominal particle sizes of 10 μm, 5–15 μm and 6–9 μm respectively, from Alfa Aesar (Ward Hill, USA). Al, Zn, Ni and Cu plates with 3.175 mm thickness were purchased from OnlineMetals (Seattle, USA). We used a water jet cutter to extract 15 × 15 × 3.175 mm plates for use as the targets for the impact experiments. Each target surface was ground and polished to 1 μm mirror finish prior to the impact experiments.

Before each impact test, particles to be launched were selected using a secondary CCD camera. For each impact, the particle diameter was extracted from the image sequence. The measured particle diameters are 14 ± 2 and 30 ± 7 μm for Al, 10 ± 2 μm for Ni, 14 ± 2 μm for Cu and 11 ± 2 μm for Zn. We have also conducted impact experiments on larger Al particles (45 μm) to resolve the particle deformation during impact with 10 ns time intervals.

Our method resolves the instant of impact with micrometer-scale spatial resolution and nanosecond-level temporal resolution. Fig. 2a shows some exemplar results taken for a 15-μm Al particle impacting an Al target a velocity of 765 m/s (±2%). The full-field video is available in the Supplementary information (Video S1) and has a field of view of 637 × 478 μm. At this velocity, the particle rebounded. Flattening of the particle upon impact can be noted in the 4th and 5th snapshots. The particle is rotating in the subsequent snapshots. The circular cross section seen in the 10th snapshot is actually the contact area upon impact, which has been flattened such that it looks larger than the incoming

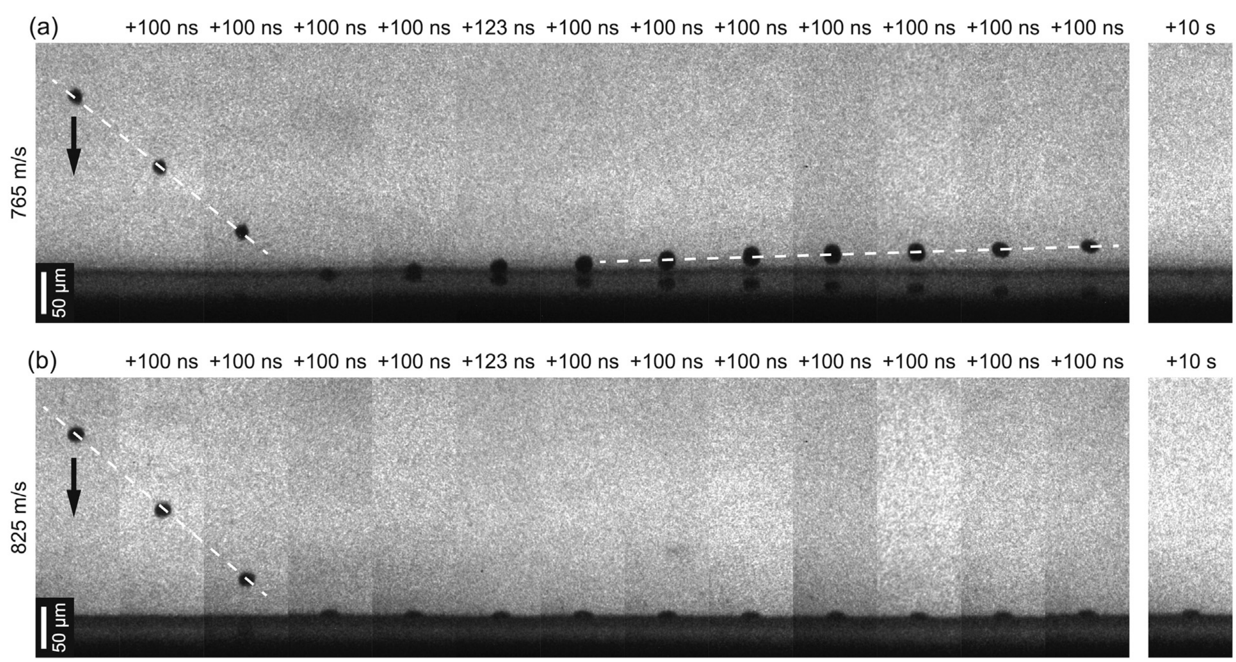

Fig. 2. In situ observation of microparticle supersonic impact. Multi-frame sequences with 5 ns exposure time showing a single Al particle impacting on an Al substrate. (a) The microparticle arrives from the top of the field of view with a speed of 765 m/s, impacts the substrate and subsequently rebounds with a speed of 35 m/s. (b) The microparticle impacts the substrate with a speed of 825 m/s and subsequently adheres to the surface. The relative delay from the initial image is shown at the top of each frame. The images are cropped from their original size to show the region of interest (see Supplementary Videos S1–S2 for a full-field view).