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AN ENVIRONMENTAL ANALYSIS OF NANOPARTICLE-ASSISTED DIFFUSION BRAZING

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Nickel Nanoparticles, Steel, Brazing, Life Cycle Analysis

ABSTRACT

Nano- and microtechnologies offer many benefits to society and promise the prospect of paradigm shifts on many technological fronts, including health care, alternative energy production, and efficient chemical processing. Current manufacturing processes for the production of nano- and microscale products, however, are energy and waste-intensive – requiring energy and creating wastes/emissions at orders of magnitude greater than traditional production. Therefore, research is needed to quantify environmental performance improvements in nano- and microproduction technologies. nickel nanoparticle (NiNP) deposition is compared with more traditional nickel phosphorus (NiP) electroplating for facilitating diffusion brazing of arrayed microfluidics. These two technologies are analyzed on a functional basis from an environmental perspective. Potential areas of improvement and future research opportunities are identified and discussed.

INTRODUCTION

Nanotechnology promises to lead to fundamental advancements in science and engineering, and an improved understanding of technological and environmental systems through inherently interdisciplinary research. The ability to create and assemble nanoscale building blocks with precisely controlled size and composition will revolutionize materials and manufacturing. Nanomanufacturing can offer lighter, stronger, and tunable materials; reductions in life cycle costs through lower failure rates; innovative devices based on new principles and architectures; and use of atomic level manipulation of structure [1]. Semiconductor, pharmaceutical, energy, chemical, and agricultural industries are being revolutionized by advances in nanotechnology [2]. In addition to novel technologies, conventional manufacturing processes are facilitated by phenomena at the nano- and microscale, e.g., atomic diffusion.

Diffusion bonding is a solid state joining technique for two dissimilar metals performed under heat and pressure, which causes atoms to move across the interface. Similarly, diffusion brazing is a transient liquid phase joining process utilizing a filler material that melts and diffuses into the parent material to form the joint. These two processes are being used extensively in packaging of MECS (micro-energy and chemical systems) devices. Both processes have been extensively used in micro-electro-chemical, aerospace, chemical, and nuclear industries [3,4]. The packaging of arrayed microchannel structures, including microreactors and heat exchangers, using stainless steel, copper, and nickel aluminide through diffusion bonding has been demonstrated [3,5,6].

Diffusion bonding cycle times are long and joint reliability can be problematic. The diffusion brazing technique can address these deficiencies and has been used for joining steel with an electroplated interlayer, including titanium [4,7], aluminum [8], copper [9], and low alloy steel [10] bases. Previous diffusion bonding and brazing work has relied on electroplating, which conventionally uses hazardous and toxic salts, requiring careful control. Research has investigated brazing filler materials for effective joining of stainless steel components to reduce cycle times and improve bond properties [11,12]. Mohankumar, et al. [13] and Philip, et al. [14] have reported that nanopowders can be used as the fillers in diffusion brazing for repair purposes of different alloys. A novel technique for bonding stainless steel surfaces has been proposed by Tiwari et al. [15]. In this technique, nickel nanopowder is mixed with a commercial binder system and applied to mating stainless steel surfaces.

While micro- and nanotechnologies are being vigorously pursued to support societal and economic development, concerns have been raised that nanotechnologies are not well-understood and may pose threats that outweigh the many benefits. Major concerns have been focused on the potential impacts on human health of nanoscale particles and the disproportionate energy and resource use and resultant wastes and emissions of nanomanufacturing. In particular, toxicological data is lacking; and nanoparticles can have greatly different attributes than the bulk material [16]. It has been shown that production of nanomaterials can require energy per unit mass several orders of magnitude greater than conventional material processing [17]. Material input requirements may be as much as five orders of magnitude greater than for bulk materials [18]. Thus, it is crucial for engineers and other decision makers to gain an understanding of new technologies in the support of sustainable product and process development.

A nickel nanoparticle-assisted diffusion brazing technique [15] is described below. Environmental impacts using the technique are predicted and compared to conventional diffusion bonding for a representative microfluidic device. Finally, future research opportunities are identified.

ARRAYED MICROCHANNEL MANUFACTURING

The environmental performance of two manufacturing process flows is investigated for a representative microchannel heat exchanger. As indicated in Figure 1, cold fluid enters the unit via two holes in one end plate and travels perpendicular to the internal plates. The fluid is heated, and travels via microchannels to the cold side, and exits through openings in the same end plate as the entering fluid. The device could find use in cooling small electrical components, for example.

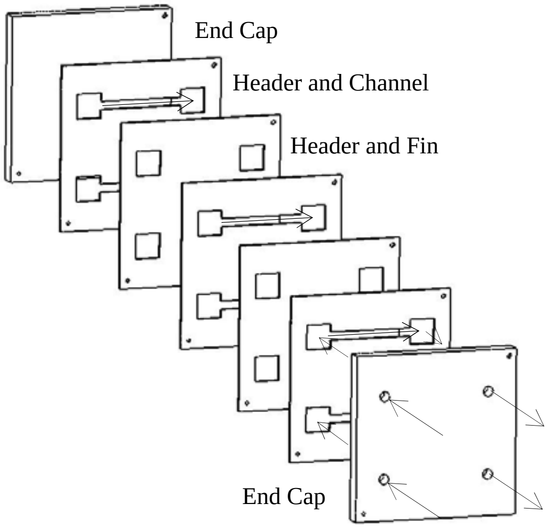


FIGURE 1. MICROLAMINATION SCHEME FOR FABRICATING A DUAL MICROCHANNEL ARRAY. [19]

The process plan for device fabrication involves machining, deburring, bond material deposition, and diffusion bonding. Detailed information for typical processes has been reported previously [20]. A common process for applying diffusion bonding material to stainless steel (AISI 316) is nickel phosphorous (NiP) electroplating. A new technique involves dispensing a nickel nanoparticle (NiNP) braze material onto the microchannel mating surfaces. Characteristics of the two bonding techniques and data for the two representative process flows are described in the next section.

Images of NiNP film, as applied to stainless steel, are shown in Figure 2 at low and high magnification. Figure 2a shows that the film is uniform with a little agglomeration. Examination of the film at higher magnification (Figure 2b) reveals that the film remains in nanoparticle form and exhibits a high surface-to-volume ratio at relatively lower temperatures, which would lend itself to more rapid diffusion into the steel.

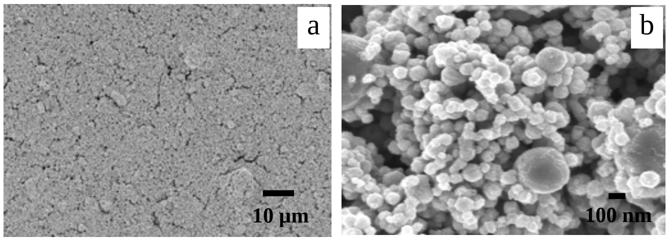


FIGURE 2. SEM IMAGES OF COATED AND DRIED (200°C, 30 min.) NiNP FILM ON STAINLESS STEEL.

SEM micrographs of the bond lines for both techniques (i.e., NiP and NiNP diffusion bonding) are shown in Figure 3. Figure 3b demonstrates that the NiNP interlayer has promising results in this application, forming a continuous bond line without microcracks or secondary phases. The secondary phase present in NiP bonding shown in Figure 3a is suspected to be intermetallics.

Void fraction analysis showed that NiNP brazing exhibits a better bond line with low void fraction (4±2%) compared to the NiP diffusion bonded sample (15±4%). A temperature depressant material, P in this case, may combine with the parent material, Fe or Ni, to form a brittle secondary phase during cooling [13]. These brittle phases can adversely affect the strength and ductility of the joint. Figure 4 shows the ultimate shear stress for both the samples.

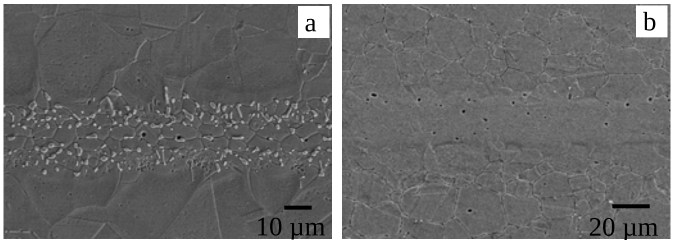


FIGURE 3. SEM IMAGES OF (a) NiP AND (b) NiNP BOND LINES [15].

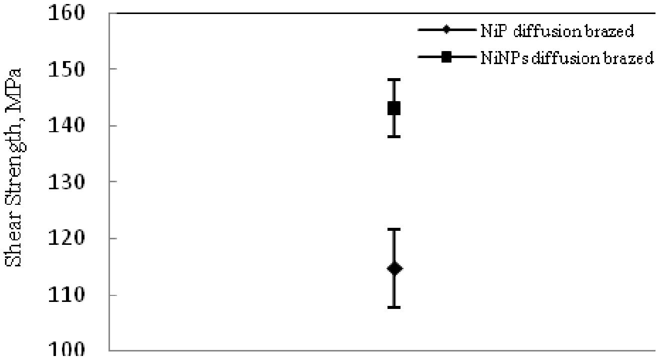


FIGURE 4. COMPARISON OF SHEAR STRENGTH FOR NiP AND NiNP DIFFUSION BONDED JOINTS.

Bond strengths of the samples were measured using single lap shear test following ASTM specifications [20]. NiNP joints have greater strength than the NiP joints, possibly due to the absence of secondary brittle phases in the bond line.

The fractured surfaces of the test samples were imaged, and the presence of ductile (cup and cone type) fracture for NiNP brazing was confirmed (Figure 5). It is apparent that the fractured surface of NiNP brazed joint has more prominent cup and cone type of fracture than the NiP joint, further demonstrating the absence of brittle secondary phases.

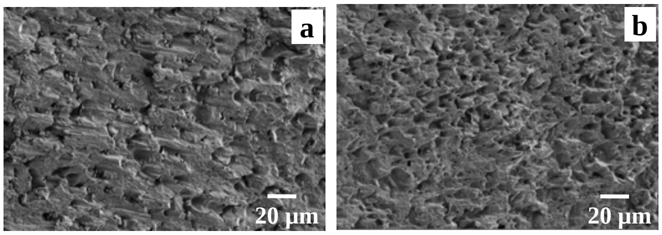


FIGURE 5. SEM IMAGES (a) NiP AND (b) NiNP BOND FRACTURE SURFACES.

From initial material property analysis, the use of nickel nanoparticles (NiNPs) in diffusion bonding appears to be a superior alternative to the diffusion bonding of nickel-phosphorus (NiP) electroplated stainless steel. The void fraction in the bond line is reduced, and the bond strength increases. The quality of the bond is also enhanced, as demonstrated by the lower variability in bond strength. Thus, while it is evident from the earlier discussion that toxicological effects of nanoparticles are not well understood, an initial screening life cycle analysis can assist in developing future nanotechnology research strategies. This approach is undertaken for nanoparticle-assisted diffusion brazing to assess its viability with respect to more conventional techniques.

ANALYSIS METHODOLOGY

To assess relative environmental impacts and identify potential future research needs, the life cycle assessment (LCA), or life cycle analysis, methodology was applied. The LCA study was facilitated using a commercially available software tool, SimaPro 7.1 [21]. In general, an LCA study is completed in four basic stages [22]: (1) Define the goal and scope, (2) Conduct inventory analysis, (3) Conduct impact assessment, and (4) Interpret results and identify opportunities for improvement.

The goal of the study was to assess the relative environmental impacts of the production of representative NiP diffusion bonded and NiNP diffusion brazed microchannel heat exchangers, and to identify opportunities for improvement. Since it is assumed bond strength will not be a critical factor impacting the life of the device in question, the functional unit of the study is one 50mm x 50mm device, with a length of about 10mm (Figure 1). The device is made of two 3mm-thick end plates, and 20 channel and 19 fin plates, each 0.1 mm thick. Each plate has two locating holes – one with a 1mm diameter and the other a 0.5mm length slot with 0.5mm radii on each end. The fluid enters/exits via four 3mm diameter holes in one end plate. The channel plates have two internal features; each is a 25mm x 2mm channel connecting two 7.5mm x 7.5mm headers. Fin plates have four 7.5mm x 7.5mm header features.

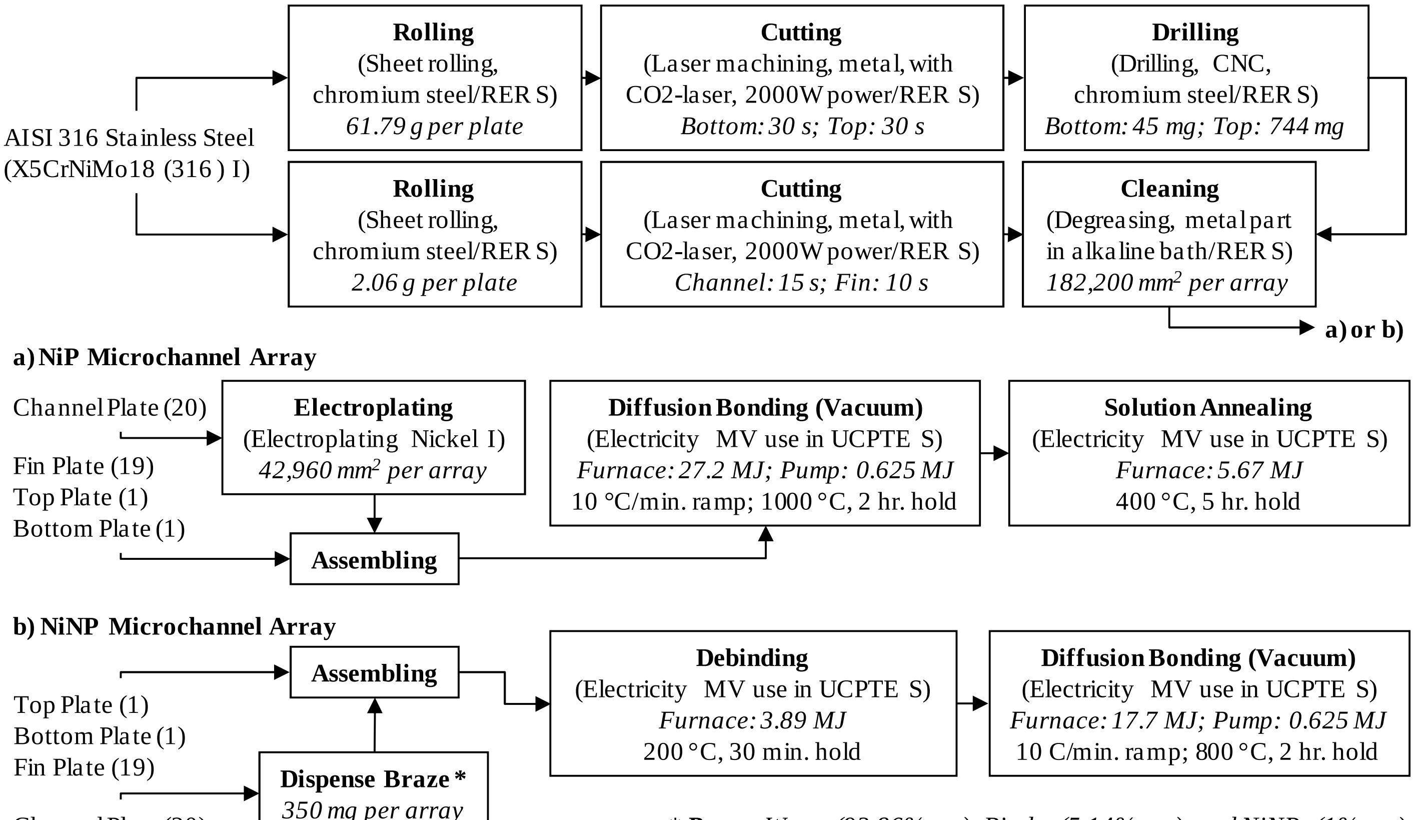
The scope of the study was cradle-to-gate, to the extent possible using LCA software and known process information. The focus was on the materials and manufacturing processes required to produce the two devices (Figure 6). Fabrication of stainless steel (AISI 316) plates is identical for both devices. The end plates are cut from 3mm thick sheet using a CO_2 laser, with holes CNC-drilled. The channel and fin plates, including internal features, are laser-cut from 0.1mm sheet. Finally, all plates are cleaned to facilitate uncontaminated bonding.

Depending on the bond type (i.e., NiP or NiNP), subsequent processing differs. The channel plates of both devices have nickel bonding material applied. In the case of the NiP device, the channel plates are electroplated using a proprietary nickel-based solution, assumed to be similar to a process in the LCA software library. Channel plates for the NiNP device are assumed to have a NiNP braze dispensed along the perimeter of the channel and plate at a 5µm thickness and 2mm width. The braze composition is reported in Table 1.

Though the composition of the binder used in the braze material is proprietary; it is known to be organic and is represented as an acrylic binder in this analysis. The NiNPs are assumed to be produced using hydrazine reduction in ethylene glycol, a low temperature process [23]. In the reported study of the process, about 380-6800 L (420-7590 kg) of ethylene glycol would be needed to produce one kilogram of NiNPs.

TABLE 1. NiNP BRAZE COMPOSITION

Wt. %	Substance	LCA Material Profile
93.86	Deionized water	Water, deionised, at plant/CH U
5.14	Binder	Acrylic binder, 34% in H2O, at plant/RER U
1.00	Nickel nanoparticles	*See Table 2*



* **Braze:** *Water (93.86% wt.), Binder (5.14% wt.), and NiNPs (1% wt.)*

FIGURE 6. MICRO HEAT EXCHANGER MANUFACTURING FLOW AND PROCESS DATA (LCA PROFILES NOTED).

Since ethylene glycol is more prone to degrade at elevated temperatures [24], it is assumed to be reused indefinitely. An equivalent mass of new solvent is added for the production of NiNP. Energy requirements could not be determined for the production of NiNPs, thus only material use is considered. In addition, the LCA software did not contain all materials to be analyzed in its library. Nickel dichloride ($NiCl_2$) was modeled as the materials used in its production [23]; hydrazine (N_2H_2) was modeled as ammonia (NH_3); and chemical reaction products were modeled as chemical waste. Data for the production of NiNPs are shown in Table 2.

Once the binders are cured and the devices are assembled, they are fused using diffusion bonding. The impacts due to diffusion bonding and the other heating processes are modeled as the impacts of electrical energy requirements of the electric furnaces and pumps. Since these processes were not available in the LCA software library, energy use was calculated and then represented as aggregated European medium-voltage electrical energy for consistency with other process profiles used. The furnace was modeled as 0.5m in each internal dimension, and considered to be constructed of 0.1m thick firebrick walls. Sixteen parts per load were assumed. The energy required to heat the parts, air in the furnace, and firebricks, as well as radiative and conductive heat loss through the walls and roof were calculated, using data shown in Table 3.

As shown in Figure 6, the NiP device undergoes diffusion bonding at 1000°C, while NiNP diffusion bonding is completed at a lower temperature (800°C). Diffusion bonding is performed in a vacuum hot press; pressure is maintained using two pumps (0.373kW, 1.48kW) operating at 75% of rated load for the 2-hour cycle.

TABLE 2. SUBSTANCES TO PRODUCE 1kg OF NiNPs [23]

Mass (kg)	Substance	LCA Material Profile
2.208	Nickel (II) chloride	Nickel I (1.000 kg); Water, deionised, at plant/CH U (1.842 kg); HCl (100%) B250 (1.242 kg) [25]
1.363	Sodium hydroxide	NaOH ETH U
1.000*	Ethylene glycol	Ethylene glycol, at plant/RER U
0.273	Hydrazine	Ammonia ETH U
0.154	Deionized water	Water, deionised, at plant/CH U
4.447	Chemical waste	Chemical waste, unspecified
* Reuse of ethylene glycol, with 1 kg added per 1kg NiNPs.		

TABLE 3. MATERIAL DATA AT 300°C [26]

	AISI 316 Stainless Steel	Air, 101 kPa	Firebrick
Density (kg/m^3)	8238	1.177	1790
Specific Heat (J/kg K)	468	1007	829
Conductivity (W/m K)			1.0
Emissivity			0.9

To improve the microstructure of the NiP device, it undergoes solution annealing at 400°C for five hours. The NiNP device requires low temperature (200°C) preheating for a half hour to facilitate debinding of the brazing material.

With the inventory analysis complete, the next step was to conduct an impact analysis. In this case, since European *ecoinvent* data were largely used, the Eco-indicator 99 method was applied to demonstrate potential relative impacts [27]. This method describes impacts in terms of Eco-indicator points – one point denotes the impact equivalent to one thousandth of the annual load of a person living in Europe.

To account for uncertainties, three weighting schemes, termed Egalitarian, Hierarchist, and Individualist archetypes, can be applied to the impact categories, which include carcinogens (C), respiratory organics (RO), respiratory inorganics (RI), climate change (CC), radiation (R), ozone layer (OL), ecotoxicity (E), acidification/eutrophication (AE), land use (LU), and minerals (M). Egalitarian and Hierarchist archetypes also account for fossil fuels (FF). The Eco-indicator 99 method considers three damage types encompassing subsets of impact categories – Human Health (C, RO, RI), Ecosystem Quality (CC, R, OL, E, AE, LU), and Resources (M, FF). Environmental impact analysis results and opportunities for improvement are discussed below.

RESULTS AND DISCUSSION

The predicted environmental impacts of NiNP and NiP device fabrication were compared using each Eco-indicator 99 archetype. In each case for all impact categories, the NiNP device outperformed the NiP device by 5-30% (Figure 7). In particular, the production of the NiNP device is predicted to outperform NiP production by at least 20% in respiratory organics (RO), radiation (R), ozone layer (OL), and land use (LU) impact categories. It performs less than 5% better in terms of minerals (M) because of the use of the same assembly components. In two cases (i.e., Egalitarian and Hierarchist), the NiNP device performs less than 10% better in terms of carcinogens (C) and ecotoxicity (E).

Relative comparisons of impact categories are reported, since impacts are measured in different units, depending on damage type. These impacts also have been converted into Eco-indicator points and aggregated to show the predicted impact for each weighting scheme for both production scenarios (Figure 8). It is interesting to note that the Individualist archetype places about ten times the level of importance on minerals than the other two types, but does not specifically account for the impacts of fossil fuels. Overall, the Individualist archetype predicts about four times the level of impact as the Egalitarian and Hierarchist archetypes. If minerals (M) are neglected, each archetype predicts similar levels of impact.

Using Egalitarian and Hierarchist archetypes, the NiNP device is predicted to have about 14% lower impact than the NiP device, while using the Individualist archetype it will have about 6% lower environmental impact. Predicted impacts in terms of damage type (i.e., Human Health, Ecosystem Quality, and Resources) for both production scenarios are shown in Figure 9. Once again, the difference in importance placed on impact categories by the three archetypes is revealed.

Interestingly, the Egalitarian and Individualist archetypes place Resources as the damage type of highest concern, while Human Health was found to be the highest concern using the Hierarchist archetype. The Individualist archetype predicts higher Human Health impacts and a lower level of Ecosystem Resource impacts than the other two archetypes. Individualist archetype Human Health impacts dominate all other impacts, except for Individualist archetype Resource impacts.

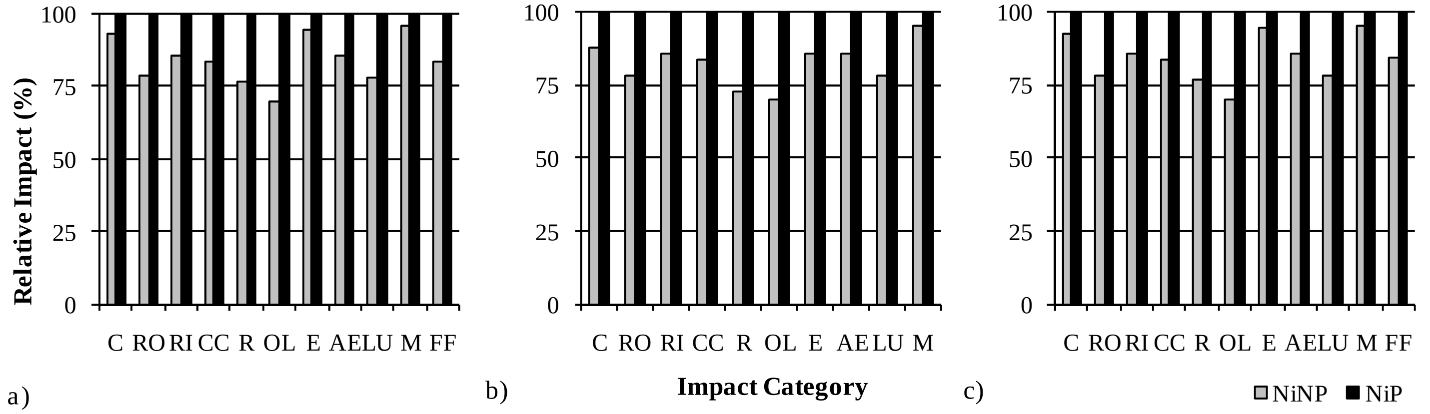


FIGURE 7. RELATIVE ENVIRONMENTAL IMPACTS FOR
a) EGALITARIAN, b) INDIVIDUALIST, AND c) HIERARCHIST ARCHETYPES.

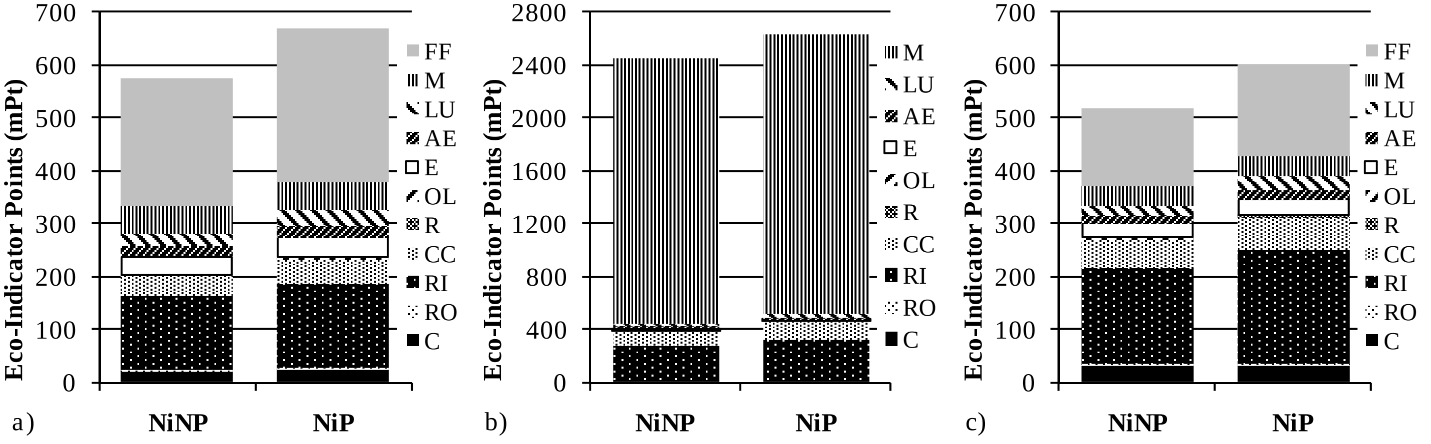


FIGURE 8. COMPARISON OF ENVIRONMENTAL IMPACTS UNDER DIFFERENT WEIGHTING SCHEMES FOR a) EGALITARIAN, b) INDIVIDUALIST, AND c) HIERARCHIST ARCHETYPES.

From Figure 8, it was seen that respiratory inorganics (RI) contributed to the majority of Human Health impacts. It is probable that this effect could to be even greater than predicted due to the health impacts of nickel nanoparticles, which are unknown and could not be accounted for in the current analysis.

To better understand the influence of each process on environmental impacts for the production of the two heat exchangers, it was decided to use system profiles, rather than unit process profiles for *ecoinvent* process data in the LCA. System profiles essentially compile upstream inputs and outputs into a single process, rather than referring to other upstream unit processes. In the latter case, the impacts of a unit process associated with all processes are aggregated, which may obscure the effects of the choice of a particular process. Capturing process impacts is of particular importance in a cradle-to-gate analysis from a manufacturing perspective, such as explored in this study.

Figure 10 shows the major contributors to the predicted environmental impacts. For the Egalitarian and Hierarchist archetypes, the energy associated with the production of stainless steel was found to be significant (not shown in the figure), and has a higher impact (18-20 mPt) than molybdenum production or sheet rolling. It is significantly lower for the Individualist archetype (8 mPt). Nickel and molybdenum production impacts are significant, as both materials are fundamental to the production of stainless steel. Nickel is also used in the electroplating process and in the braze material. The disproportionate use of nickel in the NiP device increases the associated impacts, as shown in Figure 10.

The use of the CO_2 laser to cut the stainless steel plates and the use of electricity for debinding, diffusion bonding, and solution annealing are the most impactful processes in this study. Aside from the Individualist archetype, which associates substantially greater environmental impacts with laser processing, greater impacts are expected from electricity use than laser processing for the NiP devices, while the opposite is true for NiNP devices.

These results demonstrating the higher relative impact of laser processing are consistent with studies of economic performance, which have concluded the patterning step is the dominant cost contributor in the fabrication of microlaminated devices [28, 29]. The environmental impacts for each device are identical for each major process aside from nickel and electricity – as expected due to the different bonding processes.

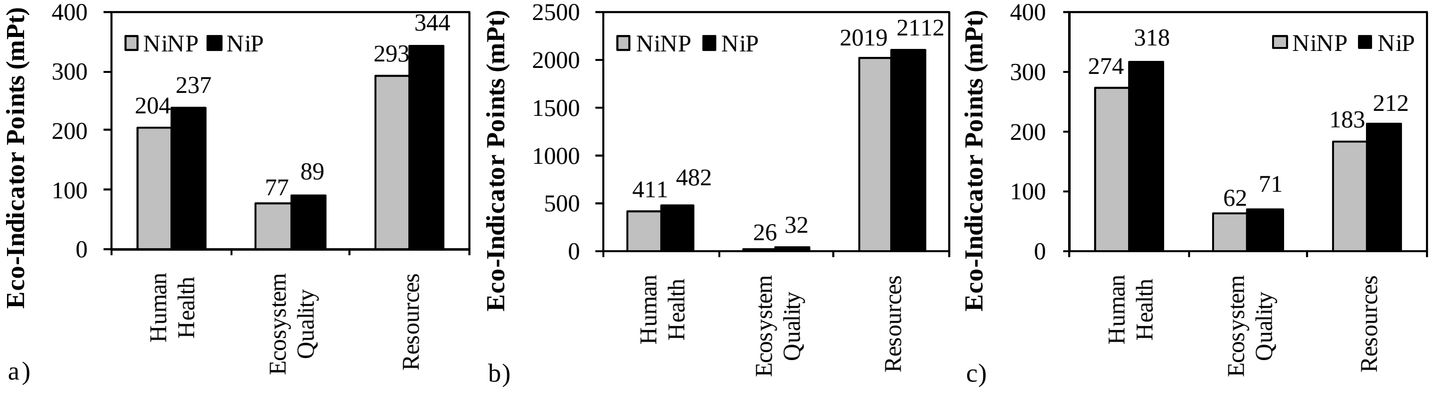


FIGURE 9. COMPARISON OF ENVIRONMENTAL DAMAGE UNDER DIFFERENT WEIGHTING SCHEMES FOR a) EGALITARIAN, b) INDIVIDUALIST, AND c) HIERARCHIST ARCHETYPES.

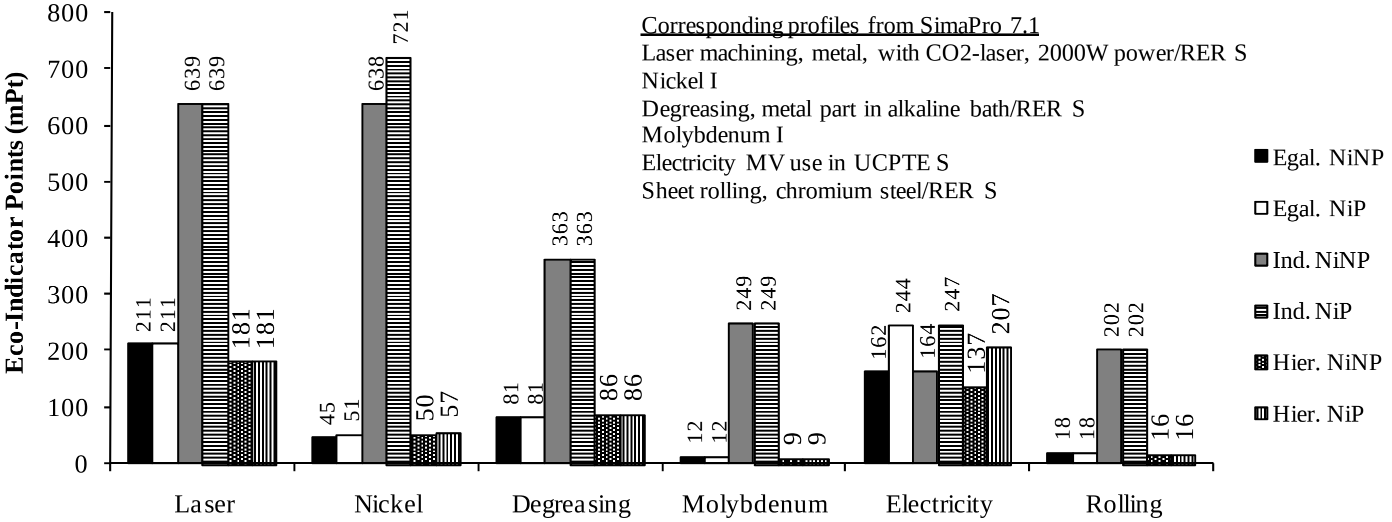


FIGURE 10. MAJOR SOURCES OF ENVIRONMENTAL IMPACTS FOR a) EGALITARIAN, b) INDIVIDUALIST, AND c) HIERARCHIST ARCHETYPES.

From the foregoing, one can conclude that the NiNP-based device outperforms the NiP device due to the lower use of nickel in brazing than in electroplating, and the reduced use of electricity facilitated by improved diffusion of nickel into the steel matrix and avoidance of solution annealing to reduce the microstructural effects of secondary phases in the bond line. In applications where bond strength is critical to the function of the device, the NiNP-bonded configuration is expected to have even more improved performance than an NiP bond.

SUMMARY AND CONCLUSIONS

A new diffusion brazing process that utilizes nickel nanoparticles (NiNPs) to assist diffusion bonding of stainless steel arrayed microchannel devices has been investigated from an environmental perspective. Cradle-to-gate environmental impacts have been compared to those of a more traditional diffusion bonding process that utilizes nickel phosphorus (NiP) electroplating. From the environmental impact analysis using Eco-indicator 99, it was concluded that the NiNP production scenario is superior to the NiP scenario, primarily due to the reduced usage of nickel and lower diffusion bonding energy requirements. Energy needs are reduced due to faster nanoparticle diffusion rates and the elimination of solution annealing to remove secondary phases in the bond line.

The environmental impact study supported previous economic studies in finding patterning to be a critical process. In these scenarios, patterning utilized a CO_2 laser to cut out microchannel plates and internal features. The laser most often ranked as the highest source of environmental impact for each impact weighting scheme. Thus, new methods of patterning microchannel devices is critical not only to reducing fabrication costs, but also vital to reducing overall environmental impacts.

In addition to process-related impacts, the impacts of the use of nickel material figured prominently in the environmental impact analysis. In this study, the impacts of nickel nanoparticles could not be determined directly due to a lack of information, but were modeled using the input materials for their production. As previous studies have shown nanomanufacturing processes to use significantly more energy and resources and produce significantly more waste on a per unit mass basis than traditional manufacturing processes, it is expected that relative process inputs and outputs would be higher for the production of nickel nanoparticles than for nickel.

Establishing a better understanding of the toxicological effects of nickel and other metal nanoparticles is of urgent importance for decision making. It is possible that potential health impacts due to NiNPs far outweigh the impacts due to laser processing, the use of electricity, or other processes considered in this analysis, and could trivialize the results of this screening study.

To protect workers in production environments from accidental exposure to nanoparticles, safer synthesis and application technologies must be developed. If nanoparticles can be produced on-demand and be encapsulated, agglomerated, or kept in solution, strides can be taken in improving the safety and viability of nano- and microscale product fabrication. Simultaneous to the development of safer nanotechnologies and manufacturing applications, new methods to facilitate the assessment of nanoparticle ecological and human health impacts must be developed. Only by providing decision makers with tools to make rapid and accurate assessments of impacts from economic, environmental, and societal perspectives will the development of sustainable nanotechnologies go forward.

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