X-ray Curing of Adhesives

By Anthony J. Berejka

The development of very high power, high-voltage accelerators makes the use of X-ray processing for industrial purposes a viable option. It has long been known that the conversion of electron beams to X-rays involves energy loses.[1] X-ray conversions range from 6-12% depending upon the accelerator voltage.[2] However, these newer high-current, high-power accelerators combined with a more fundamental understanding of X-ray effects on materials provide X-ray process throughput capabilities comparable to high-voltage (10 MV) linear accelerators.[3]

Introduction

X-rays are photon energy, as are gamma radiation, ultraviolet radiation and light.[4] Because of their shorter wavelength, X-rays directly ionize materials and create free radicals, as do electrons, without the need for initiators.[5] However, X-rays are generated from electrical sources and do not involve radioactive emissions, as does gamma radiation. X-rays, as do gamma rays, can penetrate up to ~400 mm of unit density material. Table 1 compares the properties of electron beams, X-rays and gamma radiation. Figure 1 illustrates that X-ray penetration is comparable to that of gamma radiation and exceeds that of the highest electron beam voltage (10 MV) used in commercial operations. Figure 2 illustrates the attenuation of X-rays as they are emitted from a water-cooled target (most often made of tantalum). X-ray dose-rates can be controlled by both the beam current of the impinging electrons and by the distance of the material being treated from the X-ray target.[6] X-ray dose-rates derived from commercial accelerators are in the order of kiloGrays (kGy) per minute in contrast to gamma radiation which has dose-rates of the order of kGy per hour.[7]

The commercial viability of using X-ray processing has been achieved through the development of high-current, high-voltage accelerators. For example, Radiation Dynamics Incorporated, which has more than 200 high-current, electron beam (EB) accelerators in industrial use, has developed a 300 kW, 5 MV Dynamitron® unit and its parent company, Ion Beam Applications, a 700 kW, 7 MV Rhodotron® accelerator.

Table 1

Alternative ionizing radiation sources

	Electron Beams	X-rays	Gamma Rays
Power source	Electricity	Electricity	Radioactive isotope (mainly Cobalt-60)
Power activity	Electrical on-off	Electrical on-off	5.27 year half-life
Properties	Electrons mass = 9.1×10^{-31} kg	Photons (0.30 MV) $\lambda = 4.1 \times 10^{-12}$ m	Photons (1.25 MV) $\lambda = 1 \times 10^{-12}$ m
Emission	Unidirectional (can be scanned and bent by magnets)	Forward peaked	Isotropic (cannot be controlled)
Penetration	Finite range	Exponential attenuation	Exponential attenuation
Maximum penetration (unit density)	38 mm from 10 MV entrance = exit	~ 400 mm	~300 mm
Dose-rate	360,000 kiloGray/hour 100 kGy/second	960 kiloGray/ hour 0.27 kGy/ second	10 kiloGray/hour 2.8×10^{-3} kGy/ second

X-ray Curing of Adhesive

In order to demonstrate that X-rays could cure adhesive materials, a series of commercially available adhesives were obtained that were designed for photon activation, either by ultraviolet radiation or light and based on free radical and cationic chemistry. Two opaque substrates with notably different surface characteristics and thermal properties were chosen: a ceramic and aluminum. Both substrates were ~3 mm thick and initially cut into 25.4 mm by 100 mm pieces. The ceramic used was 99.5% dense alumina (Al_2O_3) from the Superior Technical Ceramics Corporation. The aluminum pieces were etched and the ceramic wiped clean prior to adhesive application, as shown in Figure 3. Lap joints 12.7 mm by 25.4 mm were prepared.

Table 2 summarizes the differences in thermal characteristics between these two substrate materials. Such differences in thermal properties are known to create interfacial strain on bonded structures.[8] Radiation or X-ray curing is a non-thermal process. Thus, interfacial strain when bonding an insulating material, as ceramic, to a thermal conductor, as aluminum, can be reduced.[9]

A first series of test samples was cured using a 3 MV Dynamitron with a 2 cm block of aluminum placed on top of the samples, which were positioned 5 cm beneath the water cooled tantalum X-ray target, as shown in Figure 4. The aluminum block was used to hold the uncured samples in place during curing and to illustrate X-ray penetration, being too thick for use with electron beams. These were cured to 20 and 40 kGy at the adhesive interface using a dose-rate of 2 kGy per minute. Although the ceramic pieces were meticulously cut, an initial series of lap-shear bond strength tests conducted per ASTM D-1002 at a strain rate of 1.27 mm per minute (0.05 inches per minute) lead to fracture of most of the ceramic pieces, as shown in Figure 5.

To assure the integrity of the ceramic, a second series of tests was conducted using the same adhesives and substrates. However, this time the ceramic was not pre-cut, but maintained

Figure 1

Comparative EB, gamma and X-ray penetration

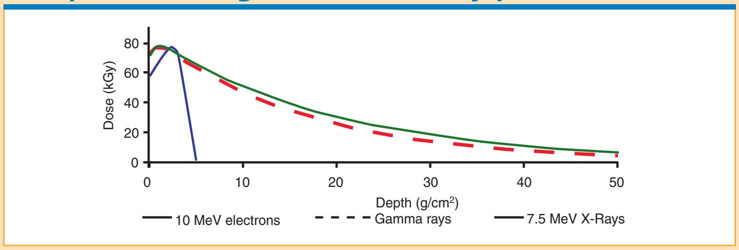

Figure 2

Forward X-ray attenuation from target

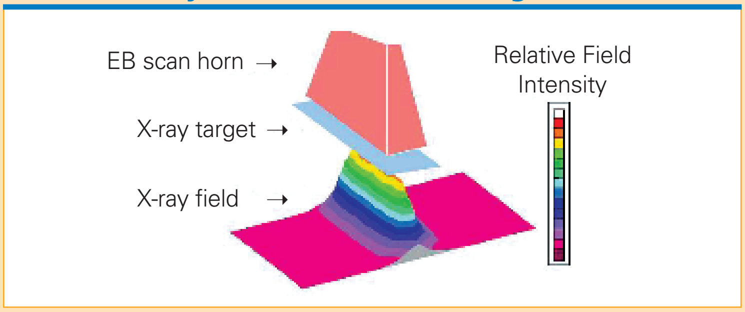

Figure 3

Application of photon curable adhesive to ceramic

Figure 4

X-ray curing of adhesive-to-ceramic lap joints

in a large piece to which the 25.4 mm wide aluminum bars were bonded. Both 25.4 mm by 25.4 mm and 12.7 mm by 25.4 mm lap joints were prepared. These were X-ray cured to 40 kGy, as shown in Figure 6.

The ceramic was then subsequently cut into widths wider than the 25.4 mm of the aluminum pieces. The bonded assemblies were then pulled in tension to determine the force required to disbond or break the specimens, as shown in Figure 7.

Table 3 presents a summary of the results obtained when X-ray curing commercial photon activated adhesives in aluminum-to-aluminum lap joints (a standard or control practice) and aluminum-to-ceramic lap joints compared with an intended use as in bonding glass to aluminum.[10]

Table 2

Substrate thermal properties

Material	Aluminum	Ceramic
Specific gravity	2.7	3.85
Thermal coefficient of expansion (x 10^{-6}/°C)	23.5	6.4
Thermal conductivity (W/m °K)	237	30

Table 3

Typical adhesive-in-shear lap joint test results

Curing method	Lap joint	Free radical chemistry	Cationic chemistry
X-ray at 40 kGy	Al-to-ceramic Al-to-Al	4.5 MPa 5.1 MPa	4.6 MPa 4.0 MPa
UV radiation	metal-to-glass metal-to-metal	~5.2 MPa cannot be done	~2.3 MPa cannot be done

Market Opportunities

Development work using X-rays to cure or crosslink materials is an innovative approach. Given the penetration of X-rays, one can envision many areas in which bonded assemblies can be adhered together even within the constraints of molds or restraining structures and so done without concern for heat transfer issues or the strains put on interfaces due to thermal processing. Aerospace assemblies needing the thermal shielding provided by ceramics can benefit from X-ray curing. Foamed ceramics are used to absorb the impact of projectiles and in bonded assemblies for military armor. Such items could be plied together using X-ray curing processes. It has been shown that photon-curable adhesive systems, designed for light or ultraviolet radiation activation, can be X-ray cured. X-ray curable adhesive materials can be simplified by elimination of costly photoinitiators. As shown in Figure 8, large scale X-ray facilities exist, which can be used for prototype development of X-ray cured bonded assemblies. This facility is now doing contract work involving X-ray processing.[11]

Figure 5

Fracture of ceramic in X-ray cured lap

Figure 6

X-ray curing of adhesive-to-ceramic lap joints

Before curing **After curing**

Figure 7

Testing of X-ray cured lap joints

Figure 8

Sterigenics X-ray processing facility, based on a 10 MV, 130 kW Rhodotron accelerator

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